UNITED STATES DEPARTMENT OF AGRICULTURE

DEPARTMENT BULLETIN No. 824

Washington, D. C. ▼ Issued June 3, 1920
Revised April, 1926

INSECT POWDER

By

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CONTENTS

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By C. C. McDonnell, *Senior Chemist,* R. C. Roark and F. B. LaForge, *Associate Chemists, Insecticide and Fungicide Laboratory,* and G. L. Keenan, *Associate Microanalyst, Microchemical Laboratory, Bureau of Chemistry*

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PURPOSE OF INVESTIGATION

Insect powder has been widely adulterated, much to the detriment of the industry. The most serious form of this adulteration has been the addition of the powdered stems of the plant to the powdered flowers, which, in some cases, has been carried to complete substitution. The work reported in this bulletin was undertaken for the purpose of devising methods for the quantitative determination of such adulteration, of determining reasonable allowances for stems and acid-insoluble ash in insect powder, and of isolating and determining the chemical nature of the active principles of insect powder.

DEFINITION

The Insecticide and Fungicide Board of the United States Department of Agriculture (*143*) [1] recognizes as insect powder an insecticide made from the powdered flower heads of the following species of Chrysanthemum:

1. *Chrysanthemum* (*Pyrethrum*) *cinerariæfolium* (Trev.) Bocc.
2. *Chrysanthemum* (*Pyrethrum*) *roseum* Web. & Mohr.
3. *Chrysanthemum Marshallii* Aschers. (synonym, *Pyrethrum carneum* M. B.).

[1] Numbers (in italic) in parentheses refer to the bibliography (p. 78). Where the letter "S" is used reference is made to the supplemental bibliography (p. 92).

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Pyrethrum is a section of the genus Chrysanthemum, which belongs to the Compositæ. The following good description of the two species of Pyrethrum commonly used for preparing insect powder is given in Bailey's Standard Cyclopedia of Horticulture, vol. 2, p. 757 (New York, 1914):

Chrysanthemum coccineum, Willd. (*Pyrethrum roseum*, Bieb., not Web. & Mohr., *P. hybridum*, Hort.). Glaubrous perennial, 1–2 ft. high; stem usually unbranched, rarely branched at the top; leaves thin, dark green, or in dried specimens dark brown; involucral scales with a brown margin; rays white or red in such shades as pink, carmine, rose, lilac, and crimson, and sometimes tipped yellow, but never wholly yellow. Caucasus, Persia.

Chrysanthemum cinerariæfolium, Vis. Glaucous perennial, slender, 12–15 in. high; stems unbranched, with a few short, scattered hairs below the flower; leaves long-petioled, silky beneath, with distant segments; involucral scales scarious and whitish at the apex. Dalmatia.

HISTORY

While all accounts of the early history of insect powder do not agree, the fact that the flowers of certain species of Pyrethrum possessed the property of killing various insects was known to the people of eastern Europe more than a century ago. Thus, according to Siedler (*257*), insect flowers have been known for more than 100 years in Dalmatia, under the name "Polvere de Pulisi."

The first published reference to the nature of insect powder was made in 1851 by Koch (*161*), who stated that the flowers of *P. roseum* and *P. carneum* yield the celebrated Persian insect powder. Another early writer on insect powder stated that its nature was kept a secret from western Europe until early in the nineteenth century, when Sumttoff (*5*), an Armenian merchant, while traveling in the Caucasian region, discovered that insect powder was made from the ground flower heads of *Pyrethrum roseum* and *P. carneum*. In 1818 Sumttoff's son began the manufacture of the powder on a large scale, and about the same time the powder was first exported in large quantities to European countries. It is said, however, that for some time before 1818 Russia had been consuming upward of 200,000 pounds annually. Browne (*38*), Riley (*223*), and several later writers give the same account of the discovery of the nature of insect powder, except that the name of the first manufacturer appears as Jumtikoff (*7*), and the first year of manufacture as 1828. Noodt (*205*), in 1858, stated that insect powder was known to the inhabitants of Transcaucasia as "guirila." In this connection it may be of interest to know that the name Buhach, applied to insect powder made from flowers grown in California, is derived from the Slavonic word Buha, which signifies a flea. The word Buhach, however, does not appear in the Slavonic language (*55*).

According to MacOwan (*184*), the nature of insect powder was made known to Russian military authorities in the Caucasus by some Tcherkess prisoners. The cantonments there swarmed with fleas that could apparently be destroyed only by a powder prepared from *Pyrethrum roseum*, the secret of which was known to the natives.

As to the discovery and history of the *Pyrethrum cinerariæfolium*, from which the Dalmatian insect powder is prepared, still less seems to be known, but it is probable that its history has been very similar to that of the Persian powder. De Visiani (*67*), in 1854, first mentioned the use of the plant as an insecticide, and Frontali (*88*),

in 1858, remarked that the powder prepared from the flowers of *P. cinerariæfolium* had been used for many years in destroying certain insects. Riley (*222*), in 1881, stated that it was impossible to obtain definite facts on the cultivation of this plant in its native home, as the inhabitants were unwilling to give information concerning a plant the product of which they wished to monopolize. Similarly, great difficulty was experienced in getting even small quantities of the seed of *P. cinerariæfolium* that had not been baked or otherwise treated to prevent germination.

Jüttner (*150*) quotes from an article which traces the discovery of the effect of the flowers of this plant on insects back to 1840. A German woman living in Ragusa, Dalmatia, picked for decoration a bunch of wild flowers, which later, as they became withered, she threw into a corner. After several weeks she noticed that many dead insects lay near the flowers. This led to the discovery that the death of the insects was due to some virtue possessed by the flowers. Thereupon she undertook the production of insect powder, which, after her death, was continued by a pharmacist of Ragusa.

According to Linke (*173*), Persian insect powder was introduced into Europe in 1846, at Vienna, by Zacherl of Tiflis. Willemot (*294*) states that the first powder of Pyrethrum was introduced into France for the destruction of household insect pests in 1850. The powder came exclusively from provinces of the Caucasus, Persia, and Dalmatia, that from the Caucasus being the best.

Insect powder was introduced into the American drug market shortly before 1860 (*1*). No figures on the production of insect flowers in the United States are available, and the total here consumed can not be accurately stated. Consumption, however, has increased tremendously in recent years. According to the committee on the drug market of the American Pharmaceutical Association (*159, 160, 191*), the importation of insect flowers and powder at the port of New York for the fiscal years 1885 to 1887, inclusive, was as follows:

TABLE 1.—*Importation of insect flowers and powder at the port of New York, 1885 to 1887*

Product	1885	1886	1887
	Pounds	*Pounds*	*Pounds*
Insect flowers	165, 505	240, 170	262, 000
Insect powder	456, 609	302, 817	335, 000
Total	622, 114	542, 987	597, 000

According to the Oil, Paint and Drug Reporter (vol. 107, p. 48), the importation of Pyrethrum, or insect flowers, from 1918 to 1924 was as follows: In 1918, 2,298,476 pounds, valued at $422,751; in 1919, 3,399,026 pounds, valued at $667,374; in 1920, 6,827,700 pounds, valued at $2,672,576; in 1921, 3,958,657 pounds, valued at $1,749,213; in 1922, 2,600,093 pounds, valued at $789,988; in 1923, 3,962,222 pounds, valued at $1,742,108; and in 1924, 2,950,269 pounds, valued at $1,316,503. Before 1914 most of the insect flowers imported came from Europe. During the World War this supply was almost completely cut off. Since 1914 Japan has been the principal source of the insect flowers shipped into the United States.

CULTIVATION AND HARVESTING OF INSECT FLOWERS

An account (*5*), written in 1856, describes the Pyrethrum plants as growing wild in the Caucasian Mountains at an elevation of from 4,500 to 6,800 feet above sea level. They blossom in June and are harvested on a dry day, when an efficient cutter can collect from 30 to 80 pounds a day. The flower heads are usually dried in the sun, although they act more powerfully when dried in the shade.

Several authors (*150, 257, 261, S13*) have described the cultivation of insect flowers in Dalmatia. According to a communication, under date of November 13, 1915, from Benjamin F. Chase, United States consul, the insect powder at Fiume is made from the blooms of a wild chrysanthemum (*Pyrethrum cinerariæfolium* Treviranus), which grows in profusion on the east side of the Adriatic Sea, from southern Croatia to Montenegro, on the Dalmatian coast, in Herzegovina and Albania. The annual production for all of this territory is from 150 to 200 metric tons. That exported is sent chiefly from Trieste, very little going out from Fiume. With the development of the trade in insect powder has come a cultivation of the wild variety. The plant grows best in rocky and barren hills with little soil, especially in limestone formations. Humid or deep soil is not favorable for its growth. Warm and dry weather is supposed to be best, not only to develop the wild plant but to give it its special insecticidal virtue. The cultivated plant does not produce well the first year, but starts the second year, and, if well cared for, continues to grow from the same root for 20 years. It begins to bloom in May, and is first harvested in early June. One hectare (2.47 acres) of the cultivated variety yields 111,100 plants, producing 2,000 kilograms (4,412 pounds) of dry blooms. The bloom is in best condition for making the powder if cut before opening, or in the "bud." It is cut off just beneath the head. After cutting, the blooms are spread on cloths and dried in the sun. When thoroughly dry, they are ground into a fine powder by revolving stones, or by crushers working vertically. The finest powder is that obtained from the "buds" or the unopened wild blooms in the region of Krivosije, Dalmatia. The second quality, that from half-opened blooms, comes from Cittavecchia, Dalmatia. The third, from the full blooms, is produced in Ragusa, Dalmatia. The "buds" used in making the first-quality powder are very small, 6 to 8 millimeters in diameter, and look like a large chamomile flower. The cultivated plants bear flower heads with a diameter of from 8 to 10 millimeters, having the rays very close together and covering the crown, being again covered by involucral bracts. Powder of the third quality is prepared from flower heads with a diameter of from 10 to 12 millimeters, almost disklike in form, many of them being without ray florets.

After the nature of insect powder became known, the cultivation of Pyrethrum was taken up in several countries. Willemot (*294*) describes the growing of *Pyrethrum roseum*, introduced into France about 1856, and Heckel (*123*) discusses the cultivation of *P. cinerariæfolium* in the botanical garden of Marseilles. Efforts to grow *C. cinerariæfolium* on a commercial scale in southern France have been fairly successful (*S16, S29*). In Switzerland also the growing of insect flowers has been encouraged and seems promising (*S10, S11, S12*).

In Germany *Pyrethrum roseum* and *P. carneum* are reported by Schenck (*242*) as growing well as early as 1859, and their cultivation there was described in detail by Pauckert (*209*) in 1866. Experiments made under the direction of the Agricultural High School on the cultivation of insect-powder-producing species of Chrysanthemum near Berlin in 1886, however, were unsuccessful (*258*). In 1912 another effort was made to grow *P. cinerariæfolium* in Germany by planting some seeds from Dalmatia in the garden of the Pharmaceutical Institute of Berlin University. The winter of 1913–14 killed most of the plants. Nevertheless Siedler (*258*) believes the cultivation of Pyrethrum near Berlin can be made profitable.

Kalbruner (*151*), in 1874, stated that in Austria *Pyrethrum roseum* and *P. carneum* were frequently seen in gardens, and Labler (*166*) in 1889 described their cultivation there. The growing of *P. cinerariæfolium* at Kroneuburg near Vienna is discussed by Kuraz (*165*).

Semenoff (*253*), in 1878, stated that in the Caucasus the production of Persian insect powder made from the flowers of *Pyrethrum roseum* and *P. carneum* amounted to about 720,000 pounds annually in 1850, but that 20 years later it had decreased to less than one-third of this quantity, owing to the competition of the Dalmatian powder. The flower heads are collected from wild plants in June and July, and are dried first in the sun and then in the shade.

Simmonds (*259*), in 1891, reported that the *Pyrethrum willemotii* (the name given to *P. cinerariæfolium* by Willemot) succeeds well in Algeria. Blin (*31*) described the cultivation of Pyrethrum at the botanical station of Rouiba, Algeria, in 1903. From 500 to 900 kilograms (1,100 to 1,980 pounds) of dry flowers per hectare (2.47 acres) are obtained, depending upon the soil and the variety of Pyrethrum.

Pyrethrum cinerariæfolium has been grown successfully in Gippsland, the southeastern district of Victoria, Australia, by Paul Kleesattel (*17*). The seeds were obtained from Zara, Dalmatia, and the plants are of the true Dalmatian type. Some powder prepared from the flowers of these plants is said to have killing properties decidedly above the average. The cultivation of Pyrethrum in Victoria is described in a Melbourne agricultural paper (*154*). Hellier (*126*) sowed some seed of the *P. Willemotii* in 1872 at Grahams Town, Cape Colony, and also distributed packets of the seed. It grew well, and its insect-destroying power was "something extraordinary." In 1883 Hellier distributed more seed, and in 1890 he reported that there were good specimens of plants at Waterford, in various places in Kaffraria, and near King Williams Town.

MacOwan (*184*), of the Cape Town Botanic Garden, points out that *Pyrethrum cinerariæfolium* thrives best on an open, dry soil, not too clayey, as both the seed and the plant are easily killed by excessive moisture. The seed, which is sown about half an inch below the surface, germinates in approximately 30 days. As soon as the plants can be handled they are placed 6 inches apart, and three months later, 1 foot apart. The flowers are produced in the second year. In the same article it is stated that the plant appears to stand the winter in sandy loam in the south of England, but has not been observed to flower freely.

In Japan the *Pyrethrum cinerariæfolium* has been grown for some time, and the manufacture of insect powder is a well-established

industry. The following information as to its cultivation there was furnished by George N. West, American consul at Kobe, under date of March 22, 1915:

The species of Pyrethrum cultivated in Japan for the manufacture of insect powder is *Chrysanthemum cinerariæfolium* (white flower, commonly called "Dalmatia"). The *C. roseum* (red flower, commonly called "Persia") is also cultivated to a small extent, not only for the same purpose, but for the beauty of its charming flowers.

None of the species of Pyrethrum are natives of Japan, but have been introduced from foreign countries. As to their introduction, it is hard to say exactly, but the following are some of the most reliable traditions:

1. During the year 1881, insect powder was first imported by one Tasaburo Shimidzu of Osaka from Bays (?) & Co., of England, through Morff & Co. of Kobe.

2. Between the years 1884 and 1885 the seeds were imported from Dalmatia and cultivated by Ei-ichiro Murakami of Yasudamura, Arita County, Wakayama Prefecture.

3. When Georg Hicotrust (?) was a consul for Austria in Japan, he at one time visited Nikko with a view of admiring the natural beauty of the place. It was autumn and the chrysanthemum flowers were blooming profusely along the sides of the road. He thought they looked like the chrysanthemum of his native country which was used for killing insects, and was surprised to see such a plant growing wild in Japan. He then imported from Austria a small quantity of the seeds which were sent to Wakayama in 1886.

4. While Professor Tamari, present director of Kagoshima Higher Agricultural and Dendrological School, was a student in the United States, he sent some seeds from the California Agricultural School and the California Agricultural Experiment Station to Komaba (near Tokyo) Agricultural College. The seeds were planted there, and were thence distributed to every part of Japan.

The plants are chiefly cultivated in the prefectures of Wakayama, Aichi, Okayama, and Hiroshima, of which the plantations in Wakayama Prefecture are most progressive. Hence the following method of plantation is principally taken from that of this prefecture:

The seed time is twice a year—spring and autumn. When the seeds are sown in the spring the flower does not open in the same year, and therefore in warm districts autumn planting is considered best. Generally in the latter part of September or October a cold bed is prepared, and one-half gill of the best selected seeds, mixed with ashes of wood or fine sand, are sowed on each tsubo (about 4 square yards), covering the whole surface slightly with well-sieved fine earth or sand. Then the surface is pressed with boards and covered with straw or rice hulls, in order to keep the earth from becoming too dry. About 10 days later, when germinated, the straw or hulls are taken off. The seedlings are then thinned out 2 or 3 times, according to thickness, in order to make the intervals between them from 1½ to 2½ inches. Five or six weeks after budding, when the seedlings have grown to a height of over 1 inch, a temporary nursery is prepared, and the strong seedlings are planted at intervals of 4¼ to 5½ inches and the weak ones at intervals of 2½ to 4 inches. The following spring they are transplanted to a ridge 2 or 2½ feet high, in a dry rice field, and to a ridge of 2 feet or of ordinary height in a vegetable field. The intervals between the seedlings when transplanted should be 1 foot to 1 foot 3 inches. The fertilizers usually applied are 1,560 pounds of natural forest loam, 40 pounds of superphosphate of lime, 80 pounds of straw ashes, and 2,500 pounds of human excrement and urine for each quarter acre. There are no special methods for plowing, weeding, and irrigation. The plants are propagated not only by seedlings, as just explained, but also by dividing the roots of the plants and transplanting them. After 4 or 5 years the plants become too old, and will not bear many flowers, making it necessary to divide their roots or to sow new seeds.

Sticky or clayey soil should be avoided. Sandy soil is preferable, because the water drains freely. The slope of a hill or reclaimed land may also be used when care is taken and it is fertilized well. No analysis of the soil is obtainable. While in the nursery, a solution of sulphate of ammonia in water is applied, in addition to the fertilizers already mentioned. After transplanting in the spring, the same solution is applied. The superphosphate of lime is also widely used. No manganese salt is applied as fertilizer, but most of the soil in Japan contains a small quantity of manganese.

The flowers are generally harvested during the latter part of May or in June, during which period they are picked over four or five times. When harvested

before the flowers are opened fully, the crop is comparatively small, but if harvested after full bloom, the strength of the powder for killing insects is lessened. Care should be taken to select the time of harvesting. The proper time is when the flower petals are fully opened, until their ends are on a level with the top of the calyx and the pollen is falling. Old men and women, carrying hand baskets, can easily pick the flowers by holding them between the middle and fore fingers, and by pressing down at the top of the flower with the thumb. By shaking up and down, the flowers are soon separated from the stem. A woman generally can pick the flowers at the rate of about 40 to 50 pounds during the day.

In some districts a simpler method of harvesting is adopted. At the time of full bloom, the flowers, together with their leaves and stems, are reaped about twice, with a tool like a short-handled rake with comb-shaped teeth, and dried in the sun. It should be added that powder made from flowers picked by this comb-toothed instrument is either inferior or no good, for the reason that flowers in all stages of blooming are thereby picked, whereas to obtain the full strength flowers must be picked at exactly the right stage of blooming which can be done only by hand.

The flowers picked are spread on straw mats and dried in a sunny place, exposed to the wind. Then they are moved to a drying room, which should have a good draft. Shelves, similar to those used in sericulture, are made by laying down rush screens or old newspapers on the top, where the flowers are arranged thinly and turned over twice or more a day. A few days later, when dried to a certain extent, they may be spread out thicker than at first. The drying is finished in 6 or 7 days. When there is no drying room, they are dried entirely in the sun. By adopting this system, a much greater quantity can be dried in a shorter time, although it is inferior in quality compared with that dried in the shade. Drying in the sun takes only three days in fine weather. If the drying takes too long, the flowers lose their strength. Drying has reached the proper stage when the flowers can be roughly powdered by breaking them into small pieces upon rubbing with the thumb and forefinger. Artificial heat is also applied at large factories, the standard of the heat being 150° F.

In addition to the districts named by West, the provinces of Ki-i and Mikawa are mentioned by Fujitani (*89*) as supplying flowers. Herrera (*129*) states that the *C. cinerariæfolium* grows well in Mexico.

Efforts to introduce the cultivation of Pyrethrum into this country were made by the United States Department of Agriculture as early as 1859. In that year Bishop (*30*) reported that 250 plants of *Pyrethrum caucasicum* were in the course of cultivation in the Experimental and Propagating Garden at Washington, D. C. Markoe (*188*) describes the growing of *P. roseum* by Asa Gray in the Cambridge, Mass., Botanical Garden from seeds distributed by the Government in 1859. The seeds yielded by the American-grown plants were sown but did not germinate. The root stocks of the old plants, however, threw up shoots in the second year. Gray was of the opinion that the cultivation of the plant could be made profitable in this country. In 1860 Abel (*1*) stated that he was informed by persons receiving some of this seed that the plants were in a flourishing condition. In the eighties the United States Department of Agriculture renewed its efforts to establish the cultivation of insect-powder-producing plants. In Washington, D. C., Riley (*222*) obtained good results. He distributed seed to correspondents in Alabama, California, Dakota, Georgia, Illinois, Indiana, Iowa, Kansas, Kentucky, Maryland, Michigan, Mississippi, Missouri, New Hampshire, New Jersey, New York, North Carolina, Ohio, Ontario, Pennsylvania, Vermont, and Virginia, but in all the States, except California, the results were unfavorable, due, apparently, largely to drought and bad seed.

On the other hand, better results were obtained by persons more familiar with growing plants. Thus Peter Henderson wrote Riley (*222*) regarding *P. roseum:* "I have grown the plant and its varieties for 10 years. It is of the easiest cultivation, either by seeds or divisions. It now ramifies into a great variety of all shades, from

white to deep crimson, double and single, perfectly hardy here, and I think likely to be nearly everywhere on this continent." *Pyrethrum roseum* was also grown successfully at Germantown, Pa., and at Archer, Fla. (*222*), from seed furnished by Riley. In 1884 Riley (*225*) reported success in growing Pyrethrum in Virginia and in Maryland, and in 1885 (*226*) in Arkansas and Florida.

King (*155*), in 1886, reported the results of efforts to grow *P. roseum* in different States. At the Connecticut Experiment Station plants from seed sown in 1884 bloomed in 1885, and then died. At the Michigan station the seasons were too cool and short for the profitable growth of this plant. The New York station reported that "the plants grew well, blossomed, and some of them matured their seed." At the Pennsylvania State College the plants did not bloom. An attempt to grow this plant in Massachusetts is described in the annual report of that station for 1889 (*101*). The seeds were sown in a hotbed, and subsequently transplanted in the field, but did not mature. In 1891 (*102*) and again in 1892 (*103*) it is reported that one row of *P. roseum* was grown in the field. Goff (*104*) planted seeds of *P. roseum* at the Geneva (N. Y.) Agricultural Experiment Station in the spring of 1887. The plants did not blossom the first season, and were counted as a failure, but in the second spring they started a vigorous growth, and bloomed profusely. The powder prepared from these New York grown flowers was just as active against flies as that from *P. cinerariæfolium* flowers grown in California. Green (*105*) in 1892 recommended *P. roseum* as an ornamental plant desirable for planting in the region of the Minnesota station. In 1890 Massey (*192*) announced his intention of trying the cultivation of *P. roseum* and *cinerariæfolium* at the North Carolina Agricultural Experiment Station, but nothing seems to have come of this.

California is the only place in the United States where the cultivation of Pyrethrum has reached commercial proportions. Coquillet (*55*) states that G. N. Milco introduced the *Pyrethrum cinerariæfolium* into California about 1870, and describes the cultivation of the plant. The quantity of the present production of insect powder in California is not known, but in 1888 it was 52 tons.[2]

Klee (*158*), of the College of Agriculture of the University of California, has carried on extensive experiments on the cultivation of Pyrethrum. At the Southern Coast Range Culture Substation of the University of California, in San Luis Obispo County, Cruickshank (*59*) in 1891 reported that Pyrethrum seed sown in the fall grew, and that the plants blossomed a little, although the year was the hardest for seven years, according to old residents. Shinn (*256*), of the same station, in 1897, reported on the cultivation of Persian insect-powder plants (apparently *P. roseum*) as follows: "Plants endure the winter and bloom freely. They do not grow rapidly, however, and the culture would probably not be profitable here." In the same report (p. 72) Pyrethrum is suggested as worthy of trial as a plant suitable for cultivation on alkali soil, but no experiments seem to have been made to test this idea. Both *P. roseum* and *P. cinerariæfolium* are mentioned as being well established in the garden of the Southern California Culture Substation, in Chino Valley. Schrenk (*248*), quoting from Semler's "Die Tropische Agrikultur," describes the cultivation of *C. cinerariæfolium* in California by Dalmatians who had settled there.

[2] Insect Life, v. 1, p. 356, 1889.

Chrysanthemum cinerariæfolium has been successfully cultivated at Madison, Wis., and at the Arlington, Va., Experimental Farm of the United States Department of Agriculture. Halsted (*112, 113*) has grown hybrids of *C. roseum* and the field daisy (*C. leucanthemum*) in New Jersey. California, however, is the only place in the United States where the cultivation of Pyrethrum has continued on a commercial scale.

SUMMARY

Insect flowers are cultivated commercially in Dalmatia, Montenegro, Japan, France, Australia, Algeria, and California. The first three countries produce nearly all the flowers that enter into international trade. In 1907, 2,882,000 pounds, and in 1908, 2,615,000 pounds of insect flowers were exported from Austria (*165*). Japan, in 1913, exported 349,225 pounds of insect flowers and 211,012 pounds of insect powder, and in 1914, 819,612 pounds of the flowers and 256,567 pounds of the powder (*251*). Exportations from Japan have increased greatly since 1914. Montenegro seems to be the only country where wild insect flowers grow abundantly enough to be of commercial importance. Even there the quantity is small, as, according to Jüttner (*150*), the whole Montenegrin production of wild flowers amounts at most to 15,000 kilograms (33,000 pounds) each year.

PREPARATION OF INSECT POWDER

One of the earliest accounts of insect powder (*5*) states that the dried insect flowers are rubbed to a coarse powder with the hand, and then ground fine in a small mill. Willemot (*294*) gives directions for pulverizing insect flowers in a mortar by simply rubbing them with a pestle.

Coquillet (*55*) describes the manufacture of Buhach from the flowers of *Chrysanthemum cinerariæfolium* in California as follows:

> Arriving at the mill the flowers (which have been thoroughly dried) are fed to a set of burr millstones, just as wheat is handled in making flour by the old process. The grist is carried by an elevator to a separator which, by proper sieves, separates the coarser particles of the grist, allowing only the finest, dustlike powder to pass through. This powder is carried by an elevator to an adjoining building, where it is put up in tin cans for the market, while the coarser particles thrown off by the separator are returned to the millstones.
>
> The flowers become heated while being reduced to powder, but the latter, in passing through a large series of elevators, loses its heat to a great degree before it is put into the cans for the market.

Slaus-Kantschieder (261), in 1913, described the preparation of insect powder in Dalmatia as follows:

> The flowers are prepared as powder in Dalmatia, as well as in Trieste. The largest Dalmatian mills, located in Sebanico, are driven with electrical power from "Krkafallen." Further, several smaller concerns in Zara, Ragusa, and upon the islands of "Mittel-Dalmatiens" carry on the grinding of the flowers. In Trieste the grinding of the chrysanthemum plants is carried on in about 10 mills, and this is the place where most of the adulteration occurs.

In the United States, in addition to Stockton, Calif., where Buhach is manufactured, insect flowers are ground on a large scale in Baltimore, Peoria, and New York, and to a smaller extent in Philadelphia, St. Louis, and other places. In most cases the older firms still use stone "chaser" mills, while the newer firms employ steel disk mills.

A "chaser" mill consists simply of a pair of millstones joined by a horizontal axis, which is connected with a vertical shaft. By means of

power the shaft is turned, and the two stones roll around, one after the other, on a heavy block of granite. These millstones, which are also of granite, are about 2½ feet in diameter by 8 inches thick, and weigh several hundred pounds each. Flowers imported into this country are received in bales done up in burlap, containing on an average about 440 pounds net each. The contents of the bales are emptied on the floor, and any large stones, which are sometimes added to give weight, removed. The flowers are then shoveled or dumped into the box which surrounds one of these stone chaser mills, where they are kept in the path of the revolving stones, which are mounted about 2 feet apart, by means of a revolving arm. The flowers are soon reduced to a fine dustlike powder, which in some mills is periodically shoveled out and in others is removed from pockets in the sides of the inclosing box. The powder is so fine that it is carried up by the air currents produced by the revolving stones, and settles into pockets provided for that purpose. The top, as well as the sides of the mill, is boxed in very tightly to keep the powder from flying everywhere. After grinding, the powder is put through a sieve or bolted, and the tailings reground. In some cases a sieve of only 40 meshes to the inch is used, whereas other firms employ 110-mesh bolting cloth.

The steel disk mill, used by some firms in grinding insect flowers, consists of a series of perforated steel disks with lugs on the edge which revolve in a corrugated cylinder at a rate of from 3,000 to 3,500 revolutions per minute. The flower heads are fed into a hopper, either by hand or automatically through a chute, and are thrown with great force against the corrugations on the inside of the cylinder by the revolving disks. The disks do not rub against each other or the cylinder; the flowers are simply cut to pieces by the force of their impact against the sharp corrugations. In a mill of this kind the cylinder opens into a large box or cloth bag of close weave. If a box is used, it must be provided with a number of cloth "chimneys," which may be supported by a wooden framework. The idea of the cloth is to hold in the fine insect powder while allowing the air, which is fanned into a very strong current by the revolving disks, to filter through.

When flowers imported from Japan are ground it is necessary first to run them through a disintegrator, which consists commonly of a mill built like an ordinary large, coarsely-grinding domestic coffee mill. Before being shipped from Japan, insect flowers are wrapped in rattan or similar material and compressed into as small a bulk as possible in a press. Ordinarily four of these little bales, each of which weighs about 100 pounds, are wrapped together in burlap with metal bands and wooden strips for shipment. The flowers are so compressed in these packages that the use of the disintegrator is necessary. From the disintegrator the flowers travel on a belt to a chute through which they fall to the floor below. An electromagnet is so arranged under the belt that particles of iron, like nails, which may be present in the bale, are removed as the flowers pass down the chute. On the floor below the flowers may be fed directly into the hopper of the disk mill, or they may be run first through a cutter, which further breaks them and expedites the final pulverization.

In either process the powder becomes quite warm in the grinding, thus losing part of its moisture, but not, apparently, any of its insecticidal constituents. This loss in moisture, together with a slight mechanical loss in the milling process, amounts to 6 or 7 per cent by weight of the flowers ground.

In grinding insect flowers it is not customary to add any material to assist the pulverization. Nor, with the exception of large stones, which may have been added to the bale, and certain bits of iron which are taken out by an electromagnet, is anything removed from the flowers as they are received. Such foreign matter as stems, either adhering or loose, sand, and dirt is allowed to remain.

In Japan the process of manufacture is as follows (*292*): The flowers are dried in the shade for one day in the summer, after which some 8 pounds are placed in a stone mortar and powdered for about 10 hours. This powder is then put through a sieve, and dried by steam heat at from 80° to 90° F. for 4 or 5 hours in a drying room. When well dried it is packed in tin containers. In the sieving process from 20 to 25 per cent of the powdered flowers remain in the sieve. This refuse is not used in the best quality insect powder, but some manufacturers repowder it and mix it with the fine, good powder. Mixed with the powdered leaves and stems of the Pyrethrum plant, it is used also as a smudge for mosquitoes and flies.

EFFECT OF INSECT POWDER ON INSECTS[3]

In the account of the discovery of the nature of insect powder by Sumttoff (*5*) no details are given as to its use. It is simply stated to be one of the most active means of protection against harmful insects, "attracting them by its characteristic odor and then numbing and killing them, but to man and larger animals it is entirely harmless."

Noodt (*205*), in 1858, wrote:

> The powder has the property of numbing all insects in a short time and subsequently killing them. Strewn in the room and in the bed it is a poison for lice, fleas, bugs, flies, moths, etc. * * *.
>
> In the collection of insects it has been used for a long time not only to quickly kill them but also to protect them against other insects, and it can be recommended not only for this use but also in herbariums and other natural history collections, since ants also quickly die from it.

In 1858 Browne (*38*) recommended the trial of Persian insect powder, or a decoction of it, against the scale of orange trees (*Coccus hesperidum*), but the test does not appear to have been made. This is the first use of insect powder against a definite insect suggested in the United States, and the first time it is spoken of for use on fruit trees. About the same time Willemot (*294*) records the results of experiments on the destruction of noxious insects in France with Pyrethrum.

Glover (*99*), in 1864, described the first experiment recorded in this country on the insecticidal efficiency of Persian insect powder:

> This powder had a curious effect on some Croton roaches we were experimenting with; when sprinkled over them or placed in a circle and they made to pass over it, for a few steps they appeared very lively, but soon staggered, and after a few struggles fell over and soon ceased to live.

Saunders (*239*), in 1879, was one of the first to describe the effect of insect powder upon house flies and green aphis. About the same time Carpenter (*44*) published the results of his experiments with Pyrethrum on different insects. As a result of these experiments, he

[3] The statements given under this heading are merely quotations from the literature and are included for their historic interest. They are not to be taken as representing the present opinion of the Department of Agriculture concerning the efficacy of this product.

states that "all insects having open mouth parts are particularly susceptible to this powerful drug." Howard (*138*), in 1882, described the effect of Pyrethrum upon the heartbeat of *Plusia brassicæ* (cabbage worm).

A great amount of work has been done since 1879 by the United States Department of Agriculture and the various agricultural experiment stations in testing the efficacy of insect powder in destroying or repelling harmful insects. A review of this work, however, is foreign to the purpose of this bulletin. Those interested may consult the following list of references to recorded tests given in the bibliography (p. 78): *2, 3, 9, 11, 26, 27, 28, 35, 36, 38, 44, 46, 49, 51, 52, 53, 54, 55, 56, 61, 65, 66, 71, 72, 75, 87, 94, 96, 97, 98, 99, 100, 104, 120, 125, 128, 129, 138, 141, 148, 149, 151, 157, 165, 167, 171, 176, 178, 192, 193, 195, 200, 201, 203, 207, 208, 211, 217, 218, 221, 223, 224, 225, 226, 227, 228, 229, 230, 231, 232, 233, 241, 258, 262, 263, 264, 270, 271, 272, 290, 291, 294, S2, S26*. Other articles on the use of insect powder against different insects are listed in "Bibliography of the More Important Contributions to American Economic Entomology," published by the United States Department of Agriculture, Bureau of Entomology, Washington, 1890–1905, "Index to the Literature of American Economic Entomology," published by the American Association of Economic Entomologists, 1917, and "Le pyrèthre insecticide de Dalmatie," by Juillet (*S20*).

A combination of pyrethrum and soap has proved a very effective insecticide (*S6, S17, S21, S23*). Kerosene extracts of pyrethrum, which are made by extracting the powdered flowers with kerosene or other light mineral oil in the proportion of 1 to 2 pounds to the gallon, have been extensively sold in the United States recently.

Some investigators claim that the powder must be taken internally to be effective; others state that it kills by external contact. Many of the reported failures were no doubt due to the fact that a powder adulterated with powdered stems was used in the tests.

At present insect powder is used largely against bedbugs, cockroaches, ants, flies, mosquitoes, and other household insects, as well as plant lice and fleas on pet animals.

Although insect powder is an efficient insecticide against many insects, the plant from which it is made is not free from insect enemies. In 1884, Riley (*225, p. 416*) received specimens of *Macrodactylus subspinosus*, found on Pyrethrum plants in large numbers and apparently eating both leaves and flowers, and *Chauliognathus marginatus*, not so numerous and apparently attracted chiefly by the flowers. He states that a number of insects feed on Pyrethrum while it is growing. In 1890 he (*229*) again called attention to the fact that the *Macrodactylus subspinosus*, or rose chafer, devours the blossoms of the *Pyrethrum cinerariæfolium*.

EFFECT OF INSECT POWDER ON ANIMALS

Although insect powder is generally considered harmless to the higher animals, several cases where it has produced serious symptoms are recorded.

As early as 1858 Boucard (*34*) described the case of a woman, who had strewn much insect powder upon her bed, being taken with a headache, roaring in the ears, bloating of the face, pain in the stomach, nausea, sweating, and symptoms of syncope. About the

same time Von Wiggers (*8*) abstracted a report by an anonymous writer of a case in which a man and his son who had scattered Persian insect powder in their beds passed a restless night, during which they suffered from painful dreams, and the next day had bad headaches. In 1884 Riley (*225*), in speaking of the supposition that Pyrethrum has no effect on the higher animals, stated his own experience in which the fumes of the powder in a closed room intensified sleep and produced stupor. Coquillet (*55*) states that insect powder has no injurious effect upon human beings.

It appears also that there is a difference of opinion with respect to the action of Pyrethrum when taken into the stomach. Milco wrote Coquillet that a teaspoonful of the alcoholic extract of Buhach was administered to a certain person afflicted with tapeworm. The dose was repeated every hour for 10 consecutive hours, as a result of which the tapeworm was removed without injuring the patient in the least. On the other hand, Noodt (*205*) states that taken internally insect powder was inactive against the tapeworm, but against Ascarides it was effective when a concentrated infusion was used as a clyster. Likewise an injection of this powder against maggots in the outer ear passages had a remarkable effect. Tests showing anthelminthic properties in the flowers of *Pyrethrum roseum* and *carneum* are recorded by Schipulinsky (*244*) in 1854, and Frontali (*88*) records the same for the flowers of *Chrysanthemum cinerariæfolium* in 1858.

According to the Chemist and Druggist (*21*), an American doctor in 1898, through an accident to a child, found that insect powder has anthelminthic properties. In 1888 Holmes, in discussing a paper by Kirkby (*156*), reported a case from Hull, England, where a man had died from the effects of insect powder, but whether the death was due to the powder itself or to some adulterant was not determined. Schlagdenhauffen and Reeb (*246*) record the poisoning of seven persons in 1889 from 1 pound of insect powder which had been strewn in their beds. Bosredon (*33*), in 1897, recorded an instance of poisoning with insect powder. An infant, aged 11 months, playing with a cardboard box of the powder, broke the lid, which scattered the powder into the eyes, mouth, and nostrils. When medical aid arrived convulsions and vomiting had set in, the heartbeats were feeble, and the respiration slightly quickened. After carefully removing the adherent powder, an emetic of ipecacuanha produced free vomiting, and, except for slight inflammation of the conjunctiva, the patient quickly recovered. Additional cases of poisoning with insect powder are described by Mendelsohn (*194*), Ferrand (*81*), and the Chemist and Druggist (*16*). In the only fatal case the patient, a 2-year-old girl, had eaten about half an ounce of the powder. McCord, Kilker, and Minster (*S25*) report an occupational dermatitis among workers engaged in grinding insect flowers.

Certain species of Chrysanthemum are used as medicine. Eastes (*74*) states that *C. parthenium* is official in the French Codex, and is reputed to have tonic, stimulative, sudorific, diuretic, antipyretic, emenagogic, and anthelminthic properties. According to Henry (*127*), many chrysanthemums are used as medicine in China. A case of poisoning with *C. indicum* is described by Hoffmann (*135*). Remington (*215*) reports that the flowers of *C. album* and *flavum* are used in China for flatulency, and includes *Pyrethrum parthenium* in a list of drugs from Chile, although its use is not given. Stearns

(*266*) lists *C. leucanthemum* and *P. parthenium* as plants whose flowers were used in medicine by the natives of Michigan in the fifties. Sato (*236*) speaks of insect powder (made from flowers of *C. cinerariæfolium*) as being used in medicine. Riley (*225*) cites a case in which insect powder was copiously rubbed on a dog, as a result of which the animal became sick, being affected in the locomotive organs very much as insects are. Carruthers (*45*) states that *P. inodorum* is credited with producing lasting injury to the digestive organs of stock by damaging the lining of the stomach and causing death when eaten in large quantities. Coquillet (*55*) reports that horses fed upon the dried stems of the *P. cinerariæfolium* plant appeared to relish it very much, and were not injured by it. In 1880 Sayre (*240*) showed the toxic action of insect powder made from the flowers of *P. roseum* upon tadpoles. Fujitani (*89*) and Reeb (*214*) record experiments made upon frogs, fish, dogs, and other animals with what they regarded as the active principle of Pyrethrum flowers. These tests, however, were made with extracts of the flowers and after certain chemical treatment, so that the results obtained are not strictly comparable with the action of insect powder itself. Zeigler (*S34*) tested the action of the active principles of *C. cinerariæfolium* (extracted with ether and the solvent allowed to evaporate without heat) upon ants, cotton-boll weevils, frogs, turtles, guinea pigs, rabbits, and dogs. The active constituents of insect flowers were toxic to cold-blooded animals when administered by mouth as well as by injection. To warm-blooded animals, however, they were toxic only when injected intravenously.

ADULTERATION OF INSECT POWDER

Insect powder appears to have been extensively adulterated from the time it first entered into commerce.

In 1851 Koch (*161*) noted that in Transcaucasia Persian insect powder is adulterated with flowers of *Pyrethrum corymbosum* and other similar plants, and in Germany with chamomile. De Visiani (*67*), in 1854, mentioned the flowers of the common chamomile, *Anthemis cotula*, *A. arvensis*, and *Spartium junceum* as adulterants. Noodt (*205*), in 1858, stated that producers, in order to satisfy the great demand for insect powder, grind not only flowers, but also stems and leaves, thereby detracting from the quality. He reports that German merchants also were in the habit of mixing fresh consignments with old goods which had deteriorated with age. Schenck (*242*), in 1859, noted the use of German chamomile as an adulterant.

In 1861 Willemot (*294*) said that the numerous adulterations which insect powder imported into France between 1850 and 1860 had undergone prevented the public from appreciating its efficacy. He mentioned the following as having been found in various powders: Sumac powder, jalap, cockle of Levant, nux vomica, and arsenic. Abel (*1*) reports that Persian insect powder was adulterated with fleabane and chamomile flowers at the time of its introduction to the American market, shortly before 1860.

Schlotshauber (*247*), in 1862, found the Persian powder to contain a variety of *Pyrethrum corymbosum* W., *P. tenuifolium* Tenore, and a variety of *Anthemis arvensis* Linn. Landerer (*167, 168*), in 1875–1877, mentioned the following as adulterants: *Anthemis cotula*,

Chrysanthemum segetum, Matricaria parthenium. In 1875 Miller (*196*) reported that old-stock German or Roman chamomile flowers were ground with insect powder.

Lead chromate as an adulterant appears to be first mentioned by Grote (*108*) in 1880. In the same year, Kral (*164*) reported finding a number of samples colored with curcuma. Howie (*139*) and Conroy (*50*), in 1883, detected fustic as an added coloring matter in insect powder. Schwarz (*250*), in 1888, found a sample colored with an alcoholic solution of curcuma.

Mason, in discussing a paper by Kirkby (*156*), mentions having had a sample of "Dalmatian insect powder" which contained 60 per cent of sumac and 30 per cent of chamomile. Howie, in the same article, reports a large proportion of potato starch in a cheap powder.

Unger (*282, 283, 284*), 1888–1890, found the following adulterants in insect powder: Pyrethrum stems, barium chromate, lead chromate, curcuma, and *Chrysanthemum leucanthemum* (Hungariar daisy). Schrenk (*249*), in 1889, also mentioned the Hungariar daisy, sometimes known as the Russian daisy, as an adulterant He stated that starch is a very common adulterant. Beringer (*29*) in 1889, reported that insect powder brought into America wa extensively adulterated with the Hungarian daisy, and that th ground stems and leaves of the Pyrethrum plant were also used ε adulterants. Hart (*119*), in 1888, reported yellow ocher and whe starch as adulterants.

In the same year, Marpmann (*190*) reported the use of the pov dered root of *Veratrum album* as an adulterant of insect powder.

Thompson (*273*), in 1891, examined seven samples of insect powde put out by American manufacturers, two of which were adulterate with lead chromate. Verneau (*285*), in 1892, listed the adulterants of insect powder as follows: *Croton flavens, Anthemis cotula, Chrysanthemum segetum, Matricaria parthenium, Tanacetum vulgare, Chamomile romaine, Chrysanthemum leucanthemum,* and wheat starch. Jelliffe (*148*), in 1895, spoke of the extensive adulteration by means of the stems of the Pyrethrum plant, and proposed methods for the detection of stem tissue in a powder. Caesar and Loretz (*42*) report that in their examination of commercial insect powders they have noted the following adulterants: Quillaja, euphorbium, powdered whole chrysanthemum plant, quassia, powdered aloes, senna leaves, Hungarian daisy, saffron, and lead chromate.

In 1899, Huber (*142*) found two out of five brands of insect powder examined to be adulterated with ground oxeye daisy flowers. Tschirch and Oesterle (*281*) give *C. coronarium* and *Inula pulicaria* in addition to many of the flowers previously mentioned as being used as adulterants. Collin (*47*) identified a sample of "false" insect flowers as *Chrysanthemum pallens.*

Hockauf (*134*), in 1903, listed the following adulterants of insect powder: Flowers of different species of Chrysanthemum (*Chrysanthemum leucanthemum, C. corymbosum, C. inodorum, C. indicum*); different species of Anthemis (*Anthemis arvensis, A. tinctoria, A. cotula*); and *Helichrysum italicum.* In the same year Jean (*147*) stated that he had found potassium chromate and sawdust in commercial insect powder, and Haywood (*122*) gave the resu examination of 105 samples, 19 of which were color chromate in amounts ranging from 0.12 to 1.47 per c

Hanausek and Winton (*118*) give the following as adulterants: Heads of *Chrysanthemum leucanthemum, Helichrysum arenarium,* DC. (*Flores Stæchadis citrinæ,* yellow cat's paw, hour-glass weed, yellow-moth weed), and the stems and leaves of *Chrysanthemum cinerariæfolium.* H. Wippell Gadd and Sydney C. Gadd (*90*), in 1905, mentioned turmeric and chrome alum as adulterants, and gave methods for their detection. Three years later Grieb (*107*) found a sample adulterated with borax.

In 1912 Linke (*173*) mentioned the following: Pyrethrum stems, flowers of other species of Chrysanthemum, calendula flowers, curcuma, lead chromate, barium chromate, euphorbium, and quillaja bark. He stated that the last two are added to increase the aroma of a powder. In the same year Sattler (*238*) reported lead chromate as an adulterant.

Jüttner (*150*), who visited the insect-powder-producing regions of Dalmatia and Montenegro in 1912, calls attention to the extensive grinding of Pyrethrum stems in those countries. In order to make this stem powder correspond in color to the genuine insect powder, lead chromate is used, and, to increase the odor, from 1 to 2 per cent of pepper powder is sometimes added.

Siedler (*150*) mentions another substance used to color insect powder, namely, yellow wood, or the heartwood of *Chlorophora tinctoria.* He mentions also *Pyrethrum indicum, Bellis perennis, Tanacetum vulgare,* chamomile, quassia powder, pepper, powdered aloes, euphorbium powder, senna leaves, and flowers of "Margherita silvatica" as adulterants. The last name is unknown in botanical nomenclature, and Siedler considers it to be a fantastic designation for an unknown adulterant. Marguerite, however, is a common name for *Chrysanthemum frutescens,* which may be the flower meant. In a later article Siedler (*257*) stated that, compared to adulteration with stem powder, adulteration with foreign flowers is insignificant.

Slaus-Kantschieder (*261*), in 1913, stated that in Dalmatia the most usual form of adulteration of insect powder consists in grinding with the flowers a part of the flower stems called "stecco." Lead chromate, barium chromate, powdered bricks, starch, curcuma, and powdered almond shells are also mentioned by him as adulterants. Benjamin F. Chase, United States consul at Fiume, under date of December 15, 1915, reported that in Dalmatia it is customary to add 5 per cent of borax to the inferior grades of insect powder.

In addition to the powdered stems of the Pyrethrum plant, which have been the most extensive means of adulteration, other adulterants detected in the Insecticide and Fungicide Laboratory during the past 11 years have been lead chromate, potassium chromate, barium chromate, curcuma, sand, leaves of the Pyrethrum plant, and oxeye daisy flowers (*Chrysanthemum leucanthemum*).

Roark and Keenan (*S26*) have made a special study of the adulteration of insect powder with powdered oxeye daisy flowers, and have described means for its detection.

Substances that have been used to color or adulterate insect [illegible] may be classified as follows:

[illegible] Lead chromate, barium chromate, potassium chromate, cur- [illegible] the root of *Curcuma longa* L.), tustic (*wood of Chlorophora* [illegible] on (dried stigmas and tops of the styles of *Crocus sativus* L.),

Other species of flowers: Pyrethrum corymbosum W., *P. tenuifolium* Tenore, *P. indicum, Chrysanthemum segetum*, Hungarian daisy, Russian daisy, oxeye daisy (*Chrysanthemum leucanthemum*), garden daisy (flowers of *Bellis perennis* L.), German chamomile (dried flower heads of *Matricaria chamomilla* L.), Roman chamomile (dried flower heads of *Anthemis nobilis* L.), corn chamomile (*Anthemis arvensis* L.), mayweed (*Anthemis cotula* L.), feverfew (*Matricaria parthenium* L. Synonyms: *Chrysanthemum parthenium* (L.) Pers.; *Pyrethrum parthenium* Sm.), *Croton flavens*, tansy (leaves and tops of *Tanacetum vulgare* L.), calendula, marigold (dried ligulate florets of *Calendula officinalis* L.), yellow cat's paw, hourglass weed, yellow-moth weed (*Helichrysum arenarium* DC. *Flores Stæchadis citrinæ*), "Margherita silvatica" (*Chrysanthemum frutescens*), *Chrysanthemum coronarium, Inula pulicaria, Chrysanthemum pallens* ("false insect flowers"), *C. inodorum, Anthemis tinctoria, Helichrysum italicum, Anthemis cotula*, and *Spartium junceum*.

Miscellaneous adulterants: Almond shells, aloes (inspissated juice of leaves of various species of aloes), arsenic, borax, brick dust, chrome alum, "cockle of Levant," euphorbium (gum-resin from *Euphorbium resinifera* Berg.), fleabane, horseweed (leaves and tops of *Erigeron canadensis* L.), hellebore (powdered root of *Veratrum album*), jalap (dried tuberous root of *Exogonium purga* (Wend.) Benth.), nux vomica (dried, ripe seed of *Strychnos nux vomica* L.), pepper, quassia (wood of *Picrasma excelsa* (Swartz) Planchon and of *Quassia amara* L.), quillaja, soap-bark (dried bark of *Quillaja saponaria* Molina, deprived of its periderm), sawdust, senna leaves (dried leaves of *Cassia acutifolia* Delile or of *Cassia angustifolia* Vahl.), starch (potato and wheat), starch (variety not specified), stems and leaves of insect powder plant, sumac (dried fruit of *Rhus glabra* L.), and ground rice hulls.

HOW TO DETECT ADULTERATION

The methods which have been used in determining the genuineness of insect powders may be classified in three groups:

1. *Physiological.*—The powder to be tested is tried out directly on one or more species of insects, and the time necessary to produce death compared with the time in which the same quantity of a known genuine insect powder will kill the insect.

2. *Microscopical.*—Adulterants are detected by observation through the microscope, either with or without staining or other preliminary chemical treatment.

3. *Chemical.*—The ash, ether extract, and other chemical determinations are made and the results compared with the average values for genuine powders.

PHYSIOLOGICAL METHODS

Kalbruner (*151*), in 1874, was the first to record the physiological testing of insect powder. He states that 4 grains of a good insect powder sprinkled on a fly in a vial should produce stupor in 1 minute and death in 2 or 3 minutes. Testing a number of commercial powders in this manner, he found that from 15 to 30 minutes were required to kill flies. Flowers representing a number of species of plants, as well as the powdered stems and leaves of *Pyrethrum roseum* and *cinerariæfolium*, were tested in this way, and found to be worthless, as compared with genuine insect powder.

In 1876, De Bellesme (*63*), in order to show that the action of the active principle of Pyrethrum was not the mechanical one of closing the pores of an insect, sprinkled flies with insect powder and also with powdered leaves, wood, and other inert substances. Those sprinkled with the Pyrethrum powder were almost dead after 1 hour, while those left for 10 hours in the inert powders were uninjured.

In 1880, Sayre (*240*) tested the effect of the flowers of *Pyrethrum roseum* on flies and tadpoles. Unger (*282*), in 1888, while exam-

ining insect powders, made use of Kalbruner s "fly test." He, however, used a weighed quantity of powder placed on a sheet of white paper, over which the insect was imprisoned with a glass. Tested against *Blatta orientalis* (Oriental cockroach or black beetle) and *Acarus farinæ* in this manner, the powder from both the *P. cinerariæfolium* and the *P. roseum* killed the insects in about 45 minutes. About the same time, Goff (*104*) made tests with *P. cinerariæfolium* grown in California and a powder prepared from the flowers of *Pyrethrum roseum*, grown in New York from seed received from the United States Department of Agriculture, to determine the relative time required to paralyze flies. These tests indicated that the powder made from *P. roseum* was slightly more effective.

Hirschsohn (*133*), in 1890, while investigating the active constituent of insect powder, tested the activity of the powder by trying it on roaches. Two of the samples under investigation paralyzed roaches in 3 minutes. From his tests on *Blatta orientalis*, Thoms (*275*) concluded that the physiological test was surer than either the microscopical or chemical test in estimating the worth of a powder.

In 1895, Jelliffe (*148*) tested the action of insect powder on the common house fly (*Musca domestica*), the Croton bug or cockroach (*Blatta germanica*), the bedbug (*Cimex lectutarius*), and some black beetles (species undetermined).

Dowzard (*71*), in 1899, conducted some tests on cockroaches. Slaus-Kantschieder (*261*), in 1913, stated that although the microscopical and chemical methods of examination furnish a basis for the grading of insect powder, for the determination of the true value the physiological test must be made. He conducts the test as follows: About 1 gram of insect powder is placed in a 25 cc. flask. The flask is then closed and shaken well, after which the flies are introduced. If the powder is of good quality the flies will come to rest within one-half minute and die within 5 minutes. If the flies survive this period the insect powder is to be considered as of low value and old.

Kuraz (*165*) has recently tested a number of commercial samples of insect powder, as well as closed, half-closed, and open flowers, and Pyrethrum stems grown at Korneuburg, near Vienna, according to the method of Slaus-Kantschieder. The ordinary house fly (*Musca domestica*) was used in these tests, and the time noted in which the insect fell over on its back. The results are summarized in Table 2.

TABLE 2.—*Physiological examination of insect powder*[1]

Product	Time required					
	Minimum		Maximum		Average	
	Min.	Sec.	Min.	Sec.	Min.	Sec.
Commercial powder ground from wild flowers	0	40	1	45	1	09
Commercial powder ground from Montenegrin closed flowers	1	04	2	45	1	43
Commercial powder ground from Dalmatian half-closed flowers	1	00	2	22	1	34
Commercial powder ground from Dalmatian open flowers	2	03	4	27	3	00
Commercial powder	5	50	15	14	10	05
Commercial powder ground from open Dalmatian flowers	2	20	4	50	3	28
Commercial powder ground from closed Dalmatian flowers	0	45	1	55	1	18
Commercial powder ground from closed Montenegrin flowers	0	40	1	34	1	04
Commercial powder	3	52	10	00	6	02
Powdered closed flowers grown at Korneuburg, 1913	1	20	2	33	1	53
Powdered half-open flowers grown at Korneuburg, 1913	0	54	1	48	1	18
Powdered closed flowers grown at Korneuburg, 1914	0	45	1	43	1	10
Powdered half-open flowers grown at Korneuburg, 1914	0	40	1	45	1	15
Powdered open flowers grown at Korneuburg, 1914	0	40	1	28	1	02
Powdered stems grown at Korneuburg, 1914	3	05	8	49	5	16

[1] 50 tests were made on each sample.

In testing the action of insect powder against various insects Smith (*263*) and many other entomologists used different brands of commercial insect powders. Their results were comparative, as they were without samples of known purity, and their experiments are without value in showing the presence of adulterants.

MICROSCOPICAL METHODS

There has been much divergence of opinion as to the value of a microscopical examination of an insect powder in determining its genuineness. For instance, Jelliffe (*148*) concludes that "the microscope is the only possible means for detecting the presence of powdered stems in insect powder." On the other hand, Beringer (*29*), after examining genuine insect powder and the powder made from the flowers of the Hungarian daisy, says, "Microscopically no difference could be detected between the two powders." Howie (*139*) states that he finds chemical methods more exact and trustworthy than microscopical ones for detecting added fustic, chrome, and turmeric. Again, in discussing Kirkby's (*156*) paper, he says that he has little faith in microscopical observation for ascertaining the value of an insect powder, the physiological test with the black beetle being the best.

For recognizing the presence of certain adulterants, as for instance starch or starch-bearing materials, the microscopical examination is of great value, but in the powdered state flowers of certain of the Compositæ closely allied to Pyrethrum are so similar to insect flowers as to render their detection difficult. Again, although an adulterant can usually be detected qualitatively by the microscope, no exact quantitative method has as yet been devised for its determination.

Trottner (*278*) has worked out a method in which the value of an insect powder is determined by estimating the number of pollen grains in 1 milligram of the sample. His results, however, vary greatly, as shown in Table 3, which summarizes all of his reported determinations.

TABLE 3.—*Number of pollen grains per milligram of insect powder*

Product	Pollen grains in 1 milligram
Closed flowers of *C. cinerariæfolium* pulverized in a mortar	2, 881
Do	2, 159
Open flowers of *C. cinerariæfolium* pulverized in a mortar	545
Do	210
Do	151
Commercial powder ground from closed flowers (Riedel)	3, 066
Commercial powder ground from open flowers (Riedel)	158
Commercial powder ground from closed flowers (Cæsar and Loretz)	2, 255
Commercial powder ground from half-closed flowers (Cæsar and Loretz)	920
Commercial powder ground from open flowers (Cæsar and Loretz)	785
Commercial powder ground from closed flowers (Schuchardt)	4, 402
Commercial powder ground from half-closed flowers (Schuchardt)	5, 544
Commercial powder ground from open flowers (Schuchardt)	1, 319
Commercial powder (Apothecary A)	2, 071
Commercial powder ground from wild closed flowers (Apothecary B)	1, 235
Commercial powder (Apothecary C)	1, 176
Commercial powder ground from cultivated closed flowers (Apothecary D)	575
Commercial powder (Apothecary E)	550
Pulverized closed flowers of *P. roseum*	4, 721
Pulverized open flowers of *P. roseum*	2, 264
Flores Pyrethri rosei, pulvis No. 0 (Gehe and Co.)	5, 741
Flores Pyrethri rosei, pulvis No. 1 (Gehe and Co.)	3, 482

Morphology of Whole Insect Flowers

The flowers usually employed in the production of insect powder or Pyrethrum powder are derived from either the Dalmatian or the Persian insect flowers, botanically known as *Chrysanthemum cinerariæfolium* (Trev.) Bocc. and *C. roseum* Web. & Mohr., respectively. The Dalmatian flowers compose the greater part of the commercial insect powders, the Persian flowers being rarely seen in commerce at the present time. Of recent years, Japanese insect flowers have been coming into the market. According to one authority (*24*) the form known as *C. indicum*, with a yellow ray flower, is widely spread through China and Japan, while in the mountains of Hupeh occurs a white or pink rayed form, which has been named *C. morifolium*. Doctor Henry, who has collected specimens which are in the Kew herbarium, considered these two wild plants the probable progenitors of the cultivated strains. As far as histological characters are concerned, the Japanese flowers can not be distinguished from *C. cinerariæfolium*.

Siedler's (*150, 258*) morphological description of the Dalmatian flowers may be summarized as follows:

The Dalmatian flower stem is 8-sided and very hairy; receptacle slightly arched; involucre consisting of 3 rows of scales, the inner scales lanceolate and about 4 mm. long, the scales of the middle row about 6 mm. long. All of the scales have a flat inner surface, the outer surface being more or less keeled, possessing a scarious margin and covered with hairs. The whitish ray flowers measure about 15 mm. in length and 4 mm. in width, 3-toothed at the tip, the middle tooth being somewhat smaller than the other two. The disk flowers are tube-shaped and 5-toothed, possessing the typical Compositæ oil glands and containing more or less of the yellow, 3-pored, spiny pollen grains. The fruits of the ray flowers exhibit a different structure from those of the disk flowers, being somewhat flattened on the side lying next to the outer bracts, and possessing two furrows, while the inner side has three. The fruits of the disk florets consist of 4, sometimes 6, nerves. A small crown is present on all the fruits.

Collin (*47*) designated three distinct commercial varieties of Dalmatian insect flowers:

1. *Closed flowers.*—Flower heads, varying from 3 to 7 mm. in diameter, generally furnished with a very short striated peduncle. Bracts, greenish-yellow, closely appressed. Corollas of ligulate florets almost always entire; grayish-white in color, and wrinkled and shriveled over the tubular florets, so as to conceal them almost completely. Very few expanded flowers present; very few fragments of corollas, ovaries, or bracts mixed with flower heads.
2. *Half-closed flowers.*—Peduncle longer, even 4 or 5 cm. long. Flower heads full, bracts of a yellowish-gray color. Ligulate florets can usually be distinguished; tubular florets still retain their corollas more or less intact.
3. *Open flowers.*—Recognized by the size of the flower heads, many of which attain a diameter of from 9 to 11 mm. Usually completely expanded when gathered and hence few of them intact; some ligulate florets destitute of corollas and in many others the corollas of the tubular florets have been separated from the ovaries which remain attached to the receptacle. This variety contains abundant débris of the corollas and ovaries, and therefore is not as choice commercially as classes 1 and 2. The various parts of the Dalmatian flower head are as follows: Bracts, outermost thicker and shorter than the others; more strongly curved and more pointed at the apex; those from the middle row lanceolate, slightly curved; the inner scales are as long as the middle ones but thinner and spatulate in shape. All scales thickened in the center, gradually becoming thinner toward the margin, which is scarious and transparent. Ligulate florets with three teeth at the apex, central tooth smaller than the other two. Calyx with fringed or slashed margin. Ovary possesses five projecting ridges. Tubular floret corolla rather deeply 5-toothed. Peduncle channeled and hairy.

The Persian (*186*) or Caucasian insect flowers (*Chrysanthemum roseum*) differ somewhat from the Dalmatian flowers, and will be but briefly described. The flower heads somewhat resemble those of the Dalmatian variety, but are a little smaller. Ray florets, a reddish-purple tinge; involucral scales, dark and with reddish-brown edge. Fruits usually 10-ribbed. Involucre not as hairy as that of the Dalmatian flower.

SUMMARY

Dalmatian flowers.—Involucre: Imbricate and campanulate; scales matted with hairs; individual bracts slightly thickened or keeled, the inner bracts becoming thinner and more chaffy or scarious at the margin.

Ligulate florets: Yellowish-white or a light straw color; pistillate; apex 3-toothed, the middle tooth usually being shorter than the other two.

Disk florets: Perfect, deeply 5-toothed, and yellowish.

Achenes of ligulate florets: Distinctly 5-ribbed; rather more flattened and curved than achenes of disk florets and somewhat larger; possess small-toothed crown.

Achenes of disk florets: Distinctly 5-ribbed; not as curved or flattened as those of the ligulate florets; somewhat smaller; possess small-toothed crown.

Persian flowers.—Involucre: Imbricate and campanulate; scales almost glabrous; hairs numerous in depression at juncture of peduncle and receptacle; scales dark and bordered by a distinctly reddish-brown edge; inner scales more scarious than outer ones; appear to be more densely fibrous than those of the Dalmatian flower.

Ligulate florets: Tinged a rather purplish-red; pistillate; apex 3-toothed, middle tooth often somewhat longer than the other two.

Disk florets: Perfect, deeply 5-toothed.

Achenes of ligulate florets: Usually 10-ribbed; more flattened and curved than achenes of disk florets and somewhat larger.

Achenes of disk florets: Usually 10-ribbed; not as curved or flattened as those of the ligulate florets; somewhat smaller.

MICROSCOPICAL CHARACTERISTICS OF INSECT POWDER

Before taking up the general plan followed by the writers in the microscopical examination of a commercial powder, the most important work done by other investigators will be reviewed briefly.

Collin (*47*) has condensed the descriptions of the diagnostic characters for the various parts of the flower head of *Chrysanthemum cinerariæfolium*, which may be summarized as follows:

Fragments of the bracts: Epidermis striated and provided with numerous stomata, T-shaped hairs, and oil glands; under the epidermis of the central part there is a very characteristic fibrous hypoderma; the margins are very thin, and bear numerous T-shaped hairs.

Corolla of the ligulate florets: Upper (inner) epidermis characterized by being papillose over the whole surface, and by the sinuous striated cells of the lower (outer) epidermis.

Corolla of the tubular florets: Epidermis papillose near the apex but smooth over the remainder of the corolla, the latter portion consisting of regular cells containing rosette crystals of calcium oxalate.

Calyx of the tubular florets: Tissue of the calyx strengthened by the presence of numerous elongated, lignified cells. At the junction of the calyx with the ovary there is a disk composed of large, very irregular cells, with thick, lignified, pitted walls. Many of these cells contain prismatic crystals of calcium oxalate, one in each cell.

Ovary: Epidermis of the intercostal depressions is characterized by the presence of numerous oil glands and clinorhombic crystals. There is a lignified hypoderma similar to that of the lower part of the calyx. Walls of the ovary contain very large ducts filled with a brown granular secretion.

Anthers: Filaments consist of regularly arranged square cells; pollen grains tubercular, having three pores.

Style: Cells of apex papillose; those of the stigma present a scale-like arrangement.

Receptacle: Characterized by large, rounded, pitted cells.

Peduncle: Débris furnished with T-shaped hairs and oil glands.

Hart (*119*) has called particular attention to the large number of translucent particles which he found in the ray florets. They occurred in every part of the flower head, except the bracts, but were most numerous in the ray florets. When treated with osmic acid they darkened very slightly, if at all, but when treated with an alcoholic solution of alkannin they turned red, which proved them to be resinous.

Schrenk (*249*) observed that the stems of the flower heads of *Chrysanthemum cinerariæfolium* (furnishing the Dalmatian flowers) consisted of collenchyma tissue which exceeded in amount the bast and woody tissues of the fibro-vascular bundles. Therefore fragments of collenchyma cells would be present in proportion to the quantity of stems present, being very sparse in a good powder. The use of chloriodid of zinc was recommended for their detection. Schrenk found the scales of the involucre to be stiffened on the outer side and on both sides of the midrib by a coherent layer of sclerenchyma cells, many of which were elongated, having oblique or pointed ends and being joined in the manner of prosenchyma cells. These were usually found as fragments in the powder, and could be recognized by their walls pierced with narrow canals. He also observed these fragments to be much more numerous in the Persian than in the Dalmatian powder. He explained this by the fact that the greater portion of the very rigid, greenish, involucral scales (with the exception of the dark, reddish-brown scarious margin) consisted of sclerenchyma cells. Numerous hairs of a very characteristic structure were found on the outer surface and along the membranous edges of the scales of the Dalmatian flowers and on the flower stems as well. Each of these hairs consisted of a long cell with attenuated ends, placed horizontally on a 1 to 3 celled stalk rising from the epidermis. The terminal horizontal cell was bent and twisted in various ways, rather hooked at the end and forming feltlike layers, especially on the outermost scales.

Schrenk detected few hairs in the Persian flower insect powder which he examined. The flowers of *Chrysanthemum roseum* which he subsequently examined were almost entirely glabrous, with the exception of the hairs found where the stem widens into the receptacle, as well as at the base of the outermost scales. The hairs were of the same structure as those found on *C. cinerariæfolium*, only the terminal cells were much longer. He considered the papillæ covering the upper epidermis the most conspicuous among the fragments of the marginal corolla. These were not regarded as diagnostic, since the petals of other related species are similarly constructed. Stomata were remarkably numerous on the lower side of the marginal corolla. He did not find the insect flowers raised in California (Buhach), which belong to *C. cinerariæfolium*, any different in structure from flowers grown in their native country.

Kirkby (*156*) and Verneau (*285*) have called attention to one distinction which they believed might aid in identifying the Persian powder. They found the papillæ of the ray florets to be larger than those of the Dalmatian florets, thickened somewhat more at the apex, and with sides making a wider angle. Malfatti (*186*) has gone into the description of the Caucasian (*Chrysanthemum roseum*) flowers quite extensively, figuring the various parts of the flower head. Siedler (*258*) describes the physical characteristics of powders made from different parts of the flower.

Microscopical Examination of Insect Powder

Before attempting the critical examination of a commercial insect powder, the microscopist should become thoroughly familiar with the various parts of the insect flower head, in the whole as well as in the powdered condition. It has been found advantageous to separate the disk and ray florets, the achenes and bracts, grinding them separately to the average fineness of a commercial powder. A study of these powders ground from the separate parts of the flower head will enable one to readily recognize them in a commercial sample. Powders ground from closed and open flowers should also be examined. The closed-flower powder is rich in the spiny, 3-pored pollen grains, and should not contain an excessive amount of sclerenchymatous tissue. The presence of considerable sclerenchymatized tissue usually indicates very mature (open) flower heads. The powder of open flowers does not contain as much pollen, but tissues from the achene are present in appreciable quantity (Pl. IV, figs. 1 and 2).

The powder to be analyzed microscopically should be thoroughly mixed. This is best done by spreading the sample upon a sheet of white paper and mixing the powder with a spatula. Flattening the powder out upon the paper often reveals the presence of whole unground pieces of material which can be transferred to a microscope slide and examined. After mixing, a composite sample is taken from various parts of the sample. A small quantity of the powder is placed upon a microscope slide, a drop or two of distilled water added, and the cover glass adjusted. If examination of the water mount reveals the presence of any foreign starch, a small quantity of a solution of iodin in potassium iodid is drawn under the cover glass. This reagent stains blue any starchy material that may be present. For further examination a small portion of the powder should be mounted in chloral hydrate solution and gently heated over the flame. This solution serves to dissolve any starch that may be present, and clears the tissues generally. Until the microscopist has become thoroughly familiar with the Pyrethrum tissues, standard samples ground from the various parts of the flower head should be kept on hand for comparative study.

Siedler (*258*) employed phloroglucin and hydrochloric acid in detecting the presence of lignified tissues (vanillin reaction). On applying these reagents to disk-flower powder very little lignified tissue was evident, although pollen grains and fragments of papillate cells were numerous. The powder from the involucre showed a large number of woody elements, isolated vessels, thick-walled prosenchyma cells, scleroids, and pitted parenchyma cells. The short-stalked **T**-hairs were characteristic of this powder. The ray-flower

powder exhibited very little lignified tissue, but a large number of papillate fragments, the cuticle layer, and large epidermal cells, which were characterized by their striated surface. The powder from the receptacle showed small, yellowish-brown cells which did not give the vanillin reaction. Thin-walled, porous cells were numerous, also lignified prosenchyma cells and large isolated vessels.

Frequently the analyst is called upon to make an estimation of the approximate quantity of insect flower stems that may be present in a powder. The fragments of stem tissue, occurring to some extent in every insect powder, are readily distinguished from the other tissues present. The stems, when ground, invariably break up into longitudinal sections. Cross sections seldom appear in the powder. These long strands of collenchyma cells, fibers, and vascular elements have a characteristic appearance, possessing brushlike or slightly frayed ends, differing very much from the shredded appearance of the fragments of bract tissue. For the purpose of estimating the percentage of stem tissues present in a powder, it has been found convenient to make up standard powders, containing known quantities of ground stem tissues, to be used for comparison with the commercial samples. Mounting the powder in xylol often facilitates the detection of stem tissues. The patches of involucral tissue are invariably torn and coarsely shredded, and not in the long, smooth pieces characteristic of the stem tissue. The fruit (achene) tissue of the Dalmatian flowers occurs in squarish patches of short, thick-walled sclerenchyma cells, containing numerous crystals, many of them diamond-shaped. These crystals exhibit a variety of colors under polarized light, and are a means of distinguishing the Persian from the Dalmatian flowers, the Persian flowers lacking these crystals in the achene tissues.

Microscopically some differences exist between the Persian and Dalmatian powders. As has already been mentioned, the marginal papillæ are somewhat different in the two species, although this character would not be recommended as a final means for distinguishing between the two. The two varieties of flowers are much more easily distinguished from each other in the whole form than in the powder.

Histology of Powder Elements

Powder, when pure, is a golden yellow, turning bright yellow when mounted in potassium hydroxid (or other alkaline) solution. Japanese powders appear to be more yellow and aromatic than other commercial powders, and assume various greenish or ashen hues, depending upon the amount of stems present. The pure powder possesses more or less of an aromatic odor. If stems are present in appreciable quantity, the powder tends to have a characteristic sage or tealike odor.

Phloroglucin and hydrochloric acid are useful in determining the amount of lignified tissue present. Lignified tissues assume a red coloration with these reagents. (Phloroglucin solution: 0.1 gram in 10 cc. of 95 per cent alcohol. Concentrated hydrochloric acid.) Potassium iodid solution of iodin turns starch a deep blue (0.05 gram of iodin and 0.2 gram of potassium iodid in 15 cc. of water). Chloral hydrate solution dissolves starch and clears the tissues (about 5 parts of chloral hydrate in 5 parts of water).

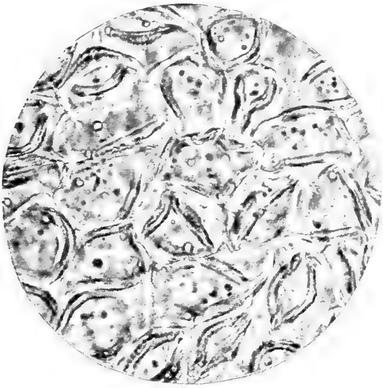


FIG. 1.—"PUCKERED" PAPILLÆ, RAY FLORET. (×290.)

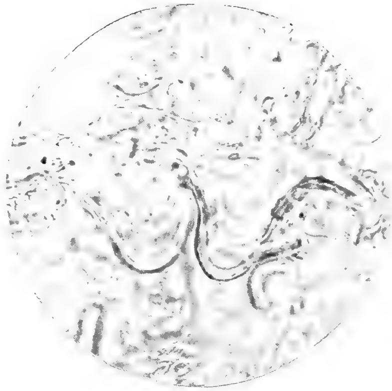


FIG. 2.—MARGINAL PAPILLÆ, RAY FLORET. (×210.)

FIG. 3.—STONE CELLS, ACHENE.

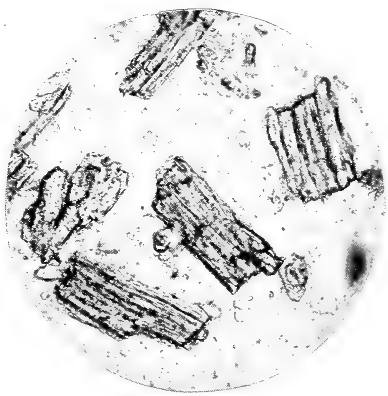


FIG. 4.—ACHENE TISSUES.

FIG. 5.—BRACT TISSUES. (×120.)

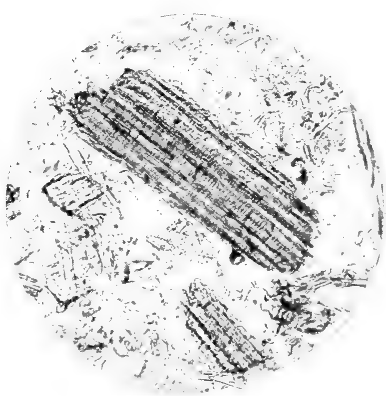


FIG. 6.—STEM TISSUES. (×84.)

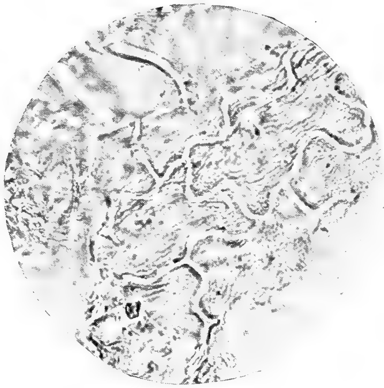


FIG. 1.—SINUOUS CELLS, RAY FLORET. (×254.)

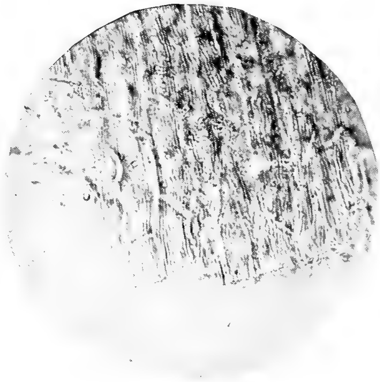


FIG. 2.—STRIATED CELLS, RAY FLORET. (×290.)

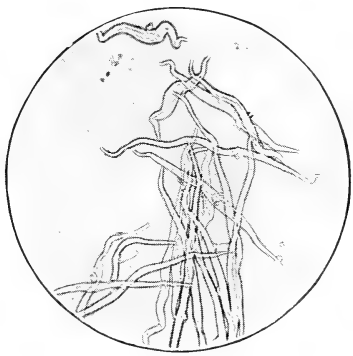


FIG. 3.—TWISTED HAIRS FROM BRACT. (×60.)

FIG. 4.—STALKED HAIR. (MAGNIFIED.)

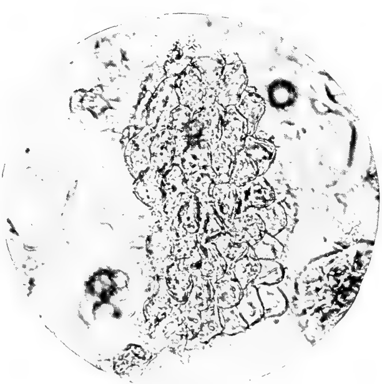


FIG. 5.—PAPILLÆ, RAY FLORET. (×140.)

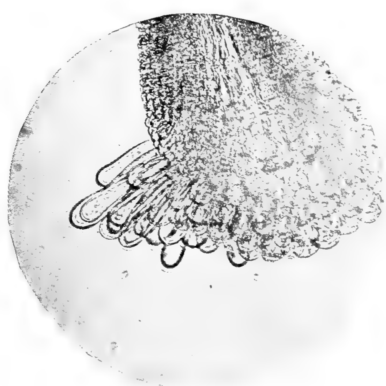


FIG. 6.—PORTION OF STIGMATIC LOBE. (×130.)

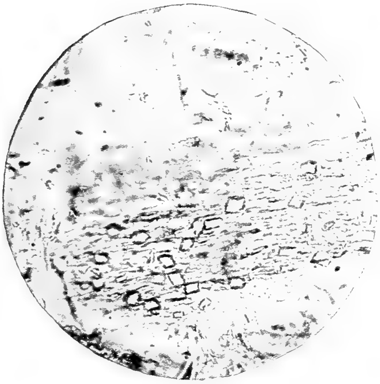


FIG. 1.—CRYSTALS, DISK FLORET. (×196.)

FIG. 2.—TOOTH, DISK FLORET. (×140.)

FIG. 3.—POLLEN GRAIN. (×290.)

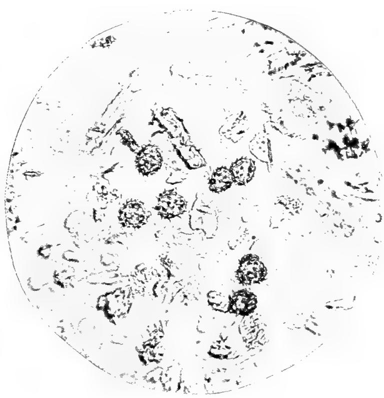


FIG. 4.—POLLEN GRAINS. (×140.)

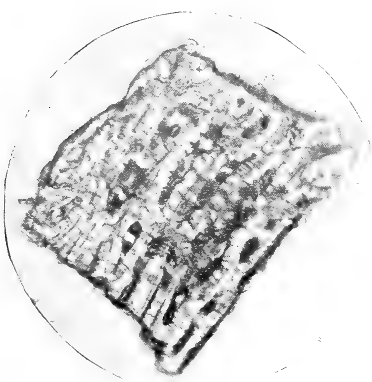


FIG. 5.—ACHENE TISSUE AND CRYSTALS. (×204.)

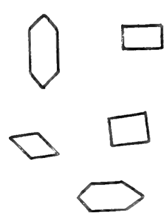


FIG. 6.—CRYSTALS FROM ACHENE. (MAGNIFIED.)

FIG. 1.—POWDER FROM OPEN FLOWERS. (×72.)

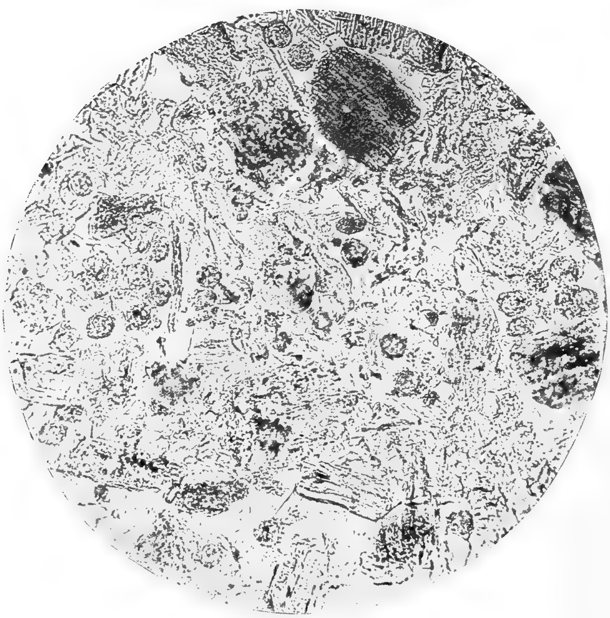


FIG. 2.—POWDER FROM CLOSED FLOWERS. (×72.)

DALMATIAN FLOWERS

1. Numerous 3-pored, spiny pollen grains (Pl. III, figs. 3 and 4).
2. Marginal papillæ and papillæ in surface view presenting a "puckered" or 3-cornered appearance (Pl. I, figs. 1 and 2).
3. Sinuous, striated cells of epidermis of ligulate florets (Pl. II, figs. 1 and 2).
4. Toothed fragments of the disk florets (Pl. III, fig. 2).
5. Shredded fragments of involucral scales, strongly lignified (Pl. I, fig. 5).
6. Attenuated and twisted horizontal cells of T-shaped hairs from the bracts (Pl. II, figs. 3 and 4).
7. Occasional strands of stem tissue, much larger than other fragments, and usually possessing roughened or fibrous ends (Pl. I, fig. 6).
8. Oil glands from the corolla and fruit. These are very seldom detected in the powder.
9. Somewhat rectangular patches of sclerenchyma tissue from the fruit, containing numerous diamond-shaped crystals exhibiting a variety of colors under polarized light. Numerous isolated stone cells are also often found in powder ground from mature flower heads (Pl. I, figs. 3 and 4; Pl. III, fig. 6).

PERSIAN FLOWERS

The diagnostic characters of the Caucasian or Persian flowers (*Chrysanthemum roseum*) in the powdered form are rather similar to those of the Dalmatian flowers. As already stated, the papillæ of the ray florets differ somewhat, and the achene tissues do not contain the crystals characteristic of the Dalmatian flower fruit. The reddish-brown scarious margins of the bracts are often more striking in the Persian powder than in the Dalmatian. Collin (*47*), who also studied the Persian flowers, has summarized the principal diagnostic characters which distinguish them from the Dalmatian flowers.

SUMMARY

Of course, the relative abundance of many of the tissues mentioned depends upon whether the powder is ground from open or closed flowers. Pollen is more abundant in closed-flower powder and sclerenchyma tissues in open-flower powder. The greater part of the pollen of closed or immature flower heads still remains in the closed "buds" or heads, the pollen not having as yet been scattered by the wind. On the other hand, the mature flower heads are practically devoid of any great quantity of pollen, but contain the mature achenes, or fruits, still attached to the receptacle, or, very often, fallen out, depending upon the ripeness of the heads. Consequently appreciably little of the achene or fruit tissues is found in closed-flower powder, whereas powder ground from open flowers is rich in the lignified tissues of the fruit and contains no pollen grains.

Stem tissues occur in all powders to some extent, although they should not be present in excessive quantity. Careful study of the stem and flower head tissues shows that there is not the slightest difficulty in distinguishing between them.

The following references in the bibliography (p. 78) deal with this subject: *23, 24, 29, 47, 69, 106, 115, 116, 117, 118, 119, 137, 148, 150, 152, 156, 162, 163, 169, 174, 186, 199, 202, 246, 249, 257, 258, 261, 278, 279, 281, 282, 285, 289.*

CHEMICAL METHODS

In all of the published work relating to the chemical analysis of insect powder the determinations have been practically confined to those of ash and of ether-soluble material, together with specific tests for turmeric, lead chromate, and other adulterants, the presence of which might be suspected. As long as the active principles of the powder were unknown, a comparison of the ash content, color, and quantity of ether extract of the sample undergoing examination with those of powders of known purity afforded the only method of judging the genuineness of a commercial powder by chemical means.

The first published analyses of insect powders are those reported in 1879 by Hilgard (*131*), who determined the ether extract of four samples with the results shown in Table 4.

TABLE 4.—*Ether-extract content of insect powder* (*Hilgard*)

Product	Ether extract
	Per cent
"Persian Insect Powder"	9.5
"Buhach" (sample grown in 1878)	6.1
"Buhach" (sample grown in 1879)	5.8
"Lyon's Magnetic Powder"	4.9

His tests of these extracts on insects showed that the quantity of the extract present was not necessarily a measure of the efficiency of the powder.

Grote (*108*), in 1880, found lead chromate in a sample of insect powder. He believes that the odor of an insect powder furnishes more evidence of its genuineness than the color. Kral (*164*), in 1880, found curcuma in a series of insect powders, but no lead chromate. Howie (*139*), in 1883, reported the results of the chemical examination of 12 insect powders. He considered the chemical method for the detection of added coloring matter as more accurate than the microscopical method. The results obtained on these 12 samples are shown in Table 5.

TABLE 5.—*Chemical examination of insect powder* (*Howie*)

Sample No.	Vendor's definition	Color	Adulterant	Ash
				Per cent
1	Insect powder	Drab	Genuine	6.2
2	do	do	do	7.1
3		Olive drab	do	6.0
4	From closed flowers	Yellowish	do	6.8
5	All closed flowers	do	do	6.2
6	Open flowers	do	do	6.2
7	Packet powder	Yellow	Chrome	10.5
8	do	do	do	9.6
9	Opt	do	do	9.2
10	All closed flowers	do	Turmeric and chrome	8.0
11	Foreign	do	Chrome	9.4
12	Dalmatian	Very yellow	Turmeric	6.0

An anonymous writer in 1884 (*13*) suggested testing insect powder with ammonia water, which would cause an artificially colored powder to turn a more or less dark brown, while an uncolored powder would change only slightly. Exposed to direct sunlight a genuine powder in the course of a few hours loses its color, according to this writer. Meyer (*202*), in 1887, proposed to test for mineral impurities in an insect powder by shaking the sample with chloroform, which would cause the powder to rise to the top of the liquid, while the inorganic substances would settle to the bottom.

In 1888 Hart (*119*) found an ash content of from 6.1 to 6.4 per cent in samples of insect powder and one of from 5.4 to 6.1 per cent in insect flowers. The ash content of the peduncles and receptacles of the flowers was 5.6 per cent. Beringer (*29*), in 1889, made the determinations given in Table 6 on flowers of the *Chrysanthemum cinerariæfolium* and of the Hungarian daisy (*C. leucanthemum*).

TABLE 6.—*Chemical analysis of flowers of C. cinerariæfolium and C. leucanthemum (Beringer)*

Product	Determination				
	Petroleum-ether extract	Ether extract	Alcohol extract	Water extract	Ash
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*
Chrysanthemum cinerariæfolium	2.49	2.85	6.57	16.70	6.50
Hungarian daisy	3.37	2.68	9.45	13.43	9.30

Beringer stated that no difference could be detected between the two powders by microscopical examination.

Unger (*282*) reports certain results of examination of Pyrethrum flowers (Table 7).

TABLE 7.—*Chemical analysis of Pyrethrum flowers and powder (Unger)*

Sample No.	Product	Determination			Remarks
		Moisture		Ash on water-free basis	
		Dried over H_2SO_4	Loss at 100°		
		Per cent	*Per cent*	*Per cent*	
1	Closed Dalmatian flowers	7.02	8.4	7.67	Much manganese present.
2	do	12.08		6.89	Do.
3	Open Dalmatian flowers	11.45		6.04	Do.
4	Insect powder	5.49		7.07	Do.
5	Closed Dalmatian flowers	10.29	10.81	7.12	Do.
6	Powdered closed Dalmatian flowers	6.76		7.56	
7	Dalmatian insect powder	5.39		6.21	Do.

Three samples of flowers, two of *Pyrethrum roseum* and one of *P. caucasicum*, grown near Berlin, were examined, the results in Table 8 being obtained.

TABLE 8.—*Chemical analysis of insect flowers grown near Berlin*

Sample No.	Product	Determination: Moisture (loss at 100°)	Determination: Ash on water-free basis	Remarks
		Per cent	*Per cent*	
1	Flowers of *P. carneum*	8.3	8.18	Much iron present, but no manganese.
2	Flowers of *P. caucasicum*	5.67	7.92	Iron (0.19 per cent) present.
3	Flowers of *P. carneum*	4.88	10.21	

Unger (*282*) reports the analyses given in Table 9 for adulterated insect powders.

TABLE 9.—*Chemical analysis of adulterated insect powders* (*Unger*)

Sample No.	Product	Ash	Remarks
		Per cent	
1	Insect powder	6.61	Stems present; almost no pollen grains.
2	do	9.7	Curcuma and stems present; pollen grains few.
3	do	7.3	Curcuma present; no manganese.
4	do	7.93	Barium chromate and many stems present; no pollen grains.
5	do	8.33	Lead chromate and many stems present; few pollen grains.
6	do	7.39	Very little manganese; curcuma present.

In another place (*282, p. 167*) Unger has recorded the results shown in Table 10.

TABLE 10.—*Chemical analysis of insect powders and flowers from Dalmatia* (*Unger*)

Sample No.	Product	Determination: Moisture	Determination: Ash on water-free basis	Remarks
		Per cent	*Per cent*	
1	Insect powder	8.56	8.33	No manganese; barium chromate and stems present.
2	do			Do.
3	do		7.3	No manganese; much iron.
4	Flowers from Spalato	8.44	6.74	Strong in manganese.

From the fact that he found no manganese in Pyrethrum stems, while it was always present in the flowers, Unger proposed to determine whether a powder was strongly adulterated with stems by testing for manganese. Thoms (*274*), in 1890, however, found manganese in the ash of *Chrysanthemum leucanthemum* and *Pyrethrum indicum*, and Siedler (*258*) showed that Pyrethrum stems are not entirely manganese-free. Appreciable quantities of manganese in Pyrethrum stems from Japan have been found by the writers.[4] Unger's test is of no value.

[4] Manganese was determined in the various grades of insect flowers and stems of both Dalmatian and Japanese origin, the following average amounts being found: Japanese stems, 0.0123 per cent; Dalmatian stems, 0.0077 per cent; Japanese closed flowers, 0.0155 per cent; Dalmatian closed flowers, 0.0096 per cent; Dalmatian open flowers, 0.0076 per cent. The manganese content of both stems and flowers varies so greatly and differs so little in the two parts of the plant that any method for estimating the amount of powdered stems in an insect powder from a determination of its manganese content is valueless.

Thoms (*274*), in 1890, reported an ash content of 6.93 per cent in whole flowers of *C. cinerariæfolium*, and one of 6.94 per cent in the same after powdering. He (*275*) would determine the value of an insect powder by a determination of its ash and petroleum-ether extract, together with a miscroscopical examination. From Dalmatian insect powder which had been dried at 100° F., Thoms obtained by extracting with petroleum ether 5.34 per cent of a brown-yellow extract (dried at 80°), which had a strong odor of insect powder. Other powders gave from 5 to 3.89 per cent. An adulterated insect powder gave only 3.83 per cent extract, without the characteristic odor. Flowers of *C. leucanthemum* (Hungarian daisy) yielded 4.02 per cent extract.

The Chemist and Druggist for March 22, 1890 (*17*) reports an ash content of 6 per cent on a sample of insect powder prepared from flowers grown in Gippsland, the southeastern district of Victoria, Australia.

Eymard (*77*), 1890, gives the analysis of Pyrethrum powder shown in Table 11.

TABLE 11.—*Analysis of Pyrethrum powder (Eymard)*

Essential oil	Small amount.
Bodies soluble in ether (5.60 per cent) composed of:	
Fatty bodies (per cent)	3. 80
Resinous bodies (per cent)	1. 80
Bodies soluble in alcohol (94.4 per cent (sic)) composed of:	
Brown resin (per cent)	4. 80
Plant albumen (per cent)	1. 75
Gummy substances (per cent)	14. 75
Inulin and soluble amidon (per cent)	8. 50
Mineral salts (per cent)	7. 88
Woody matter, by difference (per cent)	56. 72
Total (per cent)	100. 00
Mineral matter:	
Potassium chlorid (per cent)	1. 94
Calcium carbonate (per cent)	4. 15
Calcium phosphate (per cent)	0. 17
Iron and silica (per cent)	1. 62
	7. 88

Thompson (*273*) gives the results of the analysis of a number of "genuine" insect powders (Table 12).

TABLE 12.—*Analysis of "genuine" insect powder (Thompson)*

Sample No.	Color	Ash
		Per cent
1	Fawn	6. 5
2	Yellowish brown	6. 6
3	Light yellowish brown	6. 9
4	Light fawn	6. 59
5	Light yellowish brown	6. 2

Of two samples which contained lead chromate, one was deep yellow, with 12.6 per cent of ash, while the other was dark yellow, with 26.8 per cent of ash.

Gehe & Co. (*92*) give the following results of the analysis of a sample of insect powder from Dalmatia: Soluble in alcohol, 26.35 per cent; insoluble in alcohol, 56.27 per cent; water, 8.45 per cent; ash, 8.93 per cent. As they themselves state, this analysis is worthless in determining whether the sample is genuine or not.

Hill (*132*), 1894, stated that genuine Dalmatian insect powder has a greenish-yellow color, possesses a characteristic tealike odor and a slightly bitter, aromatic taste, and shows on analysis from 8 to 10 per cent of moisture, and from 6.5 to 7 per cent of ash, which is almost entirely soluble in hydrochloric acid.

Dieterich (*69*) records analyses of a number of samples of insect powders, including a determination of the maximum size of the particles in microns. He sets the following limits for a good commercial insect powder: Maximum size, 255 microns; moisture, 5.55 to 13.95 per cent. In the Erstes Dezennium der Helfenberger Annalen, 1886–95 (page 420), as the average of all determinations made on insect powders during that period, the following are given as the limiting values: Moisture (loss at 100° F.), 5.55 to 13.95 per cent; ash, 6.07 to 8.70 per cent; potassium carbonate in ash, 28 to 38.33 per cent; maximum size of particles, 109.44 to 175.50 microns.

TABLE 13.—*Chemical analysis of insect powders (Dieterich)*

Sample No.	Year	Determination			Maximum size of particles
		Moisture (loss at 100°)	Ash	K_2CO_3 in ash	
		Per cent	*Per cent*	*Per cent*	*Microns*
1		9.55	8.35	23.47	179.55
2	1896	8.25	7.65	31.57	255.15
3		11.18	7.58	30.96	151.20
1		12.12	7.38	33.80	190.35
2	1897	9.53	6.61	38.05	139.05
3		11.12	7.45	17.01	153.90
4		10.60	6.80	33.51	141.75
1		11.26	8.06	----------	148.50
2		6.95	6.40	----------	311.85
3		----------	7.13	----------	302.40
4	1900	9.93	7.56	----------	141.75
5		9.33	8.75	----------	155.25
6		----------	6.38	----------	155.25
7		7.43	6.90	----------	198.45
1		10.33	6.65	----------	198.45
2	1901	11.97	7.63	----------	145.50
3		9.28	9.94	----------	----------
4		----------	9.63	----------	----------
1		7.97	7.01	----------	202.50
2	1902	8.83	6.39	----------	135.00
3		10.52	6.16	----------	67.50
1		10.04	7.40	----------	190.35
2	1903	8.57	6.75	----------	162.00
3		9.05	7.30	----------	135.00
1	1904	8.81	6.35	----------	162.00

In the Helfenberger Annalen for 1902 (page 186), Dieterich reports the results of the determination of the ether extract of insect powders, using the method of Fromme (*85*), in which 8 grams of powder are treated with 80 grams of ether of specific gravity 0.720 at 15° C., shaken at intervals during 1 hour, then 50 grams (5 grams powder) shaken with 1 gram of water and filtered. The filter is thoroughly washed with ether, and the filtrate evaporated and weighed. The

extract of insect powders made in this way varied from 5.16 to 5.38 per cent.

Durrant (*73*) is the author of the ether extraction method that has been most generally applied in the valuation of insect powder. He proceeds as follows:

Place 100 grains of the powder in the cylinder of a glass syringe (1 ounce). The powder should be pressed down compactly on to a piece of absorbent cotton to act as a filter. Moisten with ether (0.735 sp. gr.); close the top of the syringe and macerate for 30 minutes; percolation may then proceed, the powder being repercolated with the same fluid four times, and finally washed through with sufficient ether to make up 1 fluid ounce. The resulting percolate should be of a rich yellow color; if a pronounced green color be the result the sample may be discarded at once.

This percolate should be evaporated at 200° F., and should weigh not less than 3.75 grains (=3.75 per cent), and should have the characteristic odor of the flowers * * *. Insect powder ground from selected closed flowers is sensibly free from chlorophyll, whereas traces of it (less than 0.5 per cent) will be found in powders prepared from mixed and half-open flowers, and in the foreign ground insect powders it often amounts to from 50 to 80 per cent of the total ether extract.

Durrant suggests the determination of the amount of chlorophyll when it is present in large quantities, by converting it to glucose and determining that in the regular way. He concludes: "The value of insect powder is in direct proportion to the combined amount of essential oil and soft acid resin and in inverse proportion to the amount of chlorophyll—both statements to be read together."

Francis (*84*) determined the ether extract of a powder made from "ground flowers only," one from "ground stems only," and of a "mixture of these two in equal proportion," to be as follows: Ether extract of flowers, 6.07 per cent; ether extract of mixture, 3.82 per cent; ether extract of stems, 2.25 per cent. He states, "The ether extracts in each instance had a decided green color, indicating the presence of chlorophyll."

Dowzard (*71*) estimates ether-soluble matter as follows: "Two grams of the sample are mixed with 50 cc. of ether in a stoppered cylinder, the mixture being shaken at intervals during 2 hours; 25 cc. (=1 gram powder) of the clear ethereal solution is evaporated in a tared beaker and the residue weighed." He also makes use of physiological tests: "Two grams of the sample is transferred to a beaker, a cockroach is then introduced and the number of minutes which elapse before the insect becomes stupefied are noted." He gives the results of the examination of 12 samples shown in Table 14.

TABLE 14.—*Examination of insect powder* (*Dowzard*)

Sample No.	Period of physiological test	Ether extract	Sample No.	Period of physiological test	Ether extract	Sample No.	Period of physiological test	Ether extract
	Minutes	*Per cent*		*Minutes*	*Per cent*		*Minutes*	*Per cent*
1	4	8.4	5	5	6.4	9	7	5.0
2	5	7.6	6	5	5.6	10	8	3.2
3	5	7.4	7	5	5.4	11	12	4.2
4	5	6.6	8	5	5.0	12	12	3.0

Dowzard adds: "I think the figures prove that the value of insect powder as an insecticide is in proportion to the amount of ether-soluble matter present. Of course, it is impossible to obtain exact

results with the physiological test because of the difference in size, health, etc., of the insects. In good samples of insect powder the ether extract varies between 5 and 9 per cent."

Dietze (*70*), of the firm of J. D. Riedel, Berlin, records numerous tests on insect powders (Table 15). He used powder ground by himself from closed flowers, and also commercial powders. Dietze prefers petroleum ether to ordinary ether as a solvent, as the former takes out the "active principle only," whereas ordinary ether extracts a number of other substances at the same time. In most cases the ether extract is more or less green, and has a less powerful odor than the petroleum-ether extract. Table 16 shows the results obtained upon various adulterants of insect powder.

TABLE 15.—*Chemical analysis of insect powder* (*Dietze*)

Sample No.	Determination							
	Moisture (loss at 100°)	Ash	On water-free basis					
			Soluble in—					
			Ether		Ordinary petroleum ether	Purest petroleum ether	Chloroform	Equal mixture of chloroform and ether (0.720)
			Specific gravity, 0.735	Specific gravity, 0.720				
Powders prepared from closed flowers:	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
1	7.43	7.68	9.38	7.41			7.02	6.81
2	3.42	8.26	8.59					
3	5.51	8.28	7.27					
4	8.10	7.37	8.14					
5	6.16	8.34	8.23	7.74		3.68		
6	7.41	6.70	6.44	4.35	2.50	2.61		
7	6.95	7.70	6.46	5.24	3.15	3.12		
8	4.55	7.77	6.78	5.01	2.93	2.69		
9	8.90	6.65	6.78	5.78		3.35		
10	7.23	6.75	6.71	5.01		2.83	5.26	5.17
11	9.85	7.08	7.41	4.88	2.76	2.02		
12	8.48	7.67	6.59	4.49	2.60	2.46		
13	8.06	7.49	6.12	5.92	3.03	2.97		
Minimum	3.42	6.65	6.12	4.35	2.50	2.02	5.26	5.17
Maximum	9.85	8.34	9.38	7.74	3.15	3.68	7.02	6.81
Average	7.08	7.52	7.30	5.58	2.83	2.86	6.14	5.99
Commercial powders:								
1	8.33	6.85	4.34	2.98		1.53		
2	7.76	6.58	4.66	3.15				
3	9.15	9.08	7.28	5.12		2.64		
4	7.03	8.37	5.19	4.97	2.48	2.29		2.83
5	7.63	8.14	6.09	4.52		2.64		
6	7.88	6.83	6.87	4.04	3.03	2.91	4.87	5.19
7	11.79	6.63	6.03	4.29	2.48	2.38		
8	8.68	7.36	8.68	5.17	3.47	3.29	6.03	5.86
9	9.20	6.94	8.51	6.17	3.09	2.81		
10	6.70	8.10	6.18	3.52	2.07			
11	8.62	7.45	4.41	2.41	1.60			
12	4.55	10.89	3.72	3.43	2.79			
Minimum	4.55	6.63	3.72	2.41	1.60	1.53	4.87	2.83
Maximum	11.79	10.89	8.68	6.17	3.47	3.29	6.03	5.86
Average	8.11	7.77	5.99	4.15	2.63	2.56	5.45	4.63

TABLE 16.—*Chemical analysis of insect powder adulterants (Dietze)*

Adulterant	Determination					
	Moisture (loss at 100°)	Ash	Soluble in—			
			Ether		Ordinary petroleum ether	Purest petroleum ether
			Specific gravity, 0.735	Specific gravity, 0.720		
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
Johannesblumen von Chrys. Leucanthemum	7. 59	8. 22	3. 76	3. 17	2. 46	2. 25
Flor. chamom. vgl	10. 39	12. 72	7. 51	4. 96	4. 69	4. 14
Flor. chamom. Roman	6. 78	8. 65	10. 06	5. 34	4. 09	4. 05
Flor. chamom. Calendulæ	11. 85	9. 19	8. 20	7. 59	4. 25	4. 32
Lignum quassiæ	0. 99	2. 66	0. 23	0. 21	0. 18	0. 10
Folia sennæ	10. 13	11. 06	5. 91	3. 69	3. 36	3. 39

Dietze declares that the value of an insect powder can not be determined by any of these extraction methods, whether made with ether, petroleum ether, or chloroform, and the determination of ash and moisture, but that a practical test upon insects is necessary.

Fromme (*85*), in 1900, published results for ether extract determinations of from 6 to 7 per cent on half-opened buds and from 7 to 9.5 per cent on unexpanded buds. The ether extract of pure flowers is of a golden-yellow color, while that of the stalks is of a greenish tint, thus making it easy to detect such adulteration.

Haywood (*121*) gives the results of the chemical examination of a number of commercial insect powders shown in Table 17.

TABLE 17.—*Chemical analysis of commercial insect powders (Haywood)*

Product	Determination						
	Moisture	Ash	Ether extract	Lead in ash	Chromium in ash	Barium in ash	Turmeric
	Per cent	*Per cent*	*Per cent*				
Black flag insect powder	7. 21	8. 01	8. 91	None	None	None	None.
Buhach	6. 96	9. 69	9. 09	do	do	do	Do.
Persian insect powder	5. 82	12. 02	5. 15	Present	Present	do	Do.
Pyrethrum insect powder	7. 39	7. 80	8. 38	None	None	do	Do.
Death dust for insects	6. 75	7. 74	10. 68	do	do	do	Do.
Pyrethrum powder	7. 49	11. 91	6. 68	do	do	do	Do.
Dalmatian insect powder	6. 24	8. 35	6. 43	do	do	do	Do.
Tiger insect powder	7. 68	6. 47	6. 22	do	do	do	Do.
Magic insect powder	6. 47	5. 90	6. 35	do	do	do	Do.
Insect powder	6. 87	8. 03	6. 78	do	do	do	Do.

Later, Haywood (*122*) gave the results of the examination of commercial insect powders for the presence of chromates. Chromium was determined quantitatively in the ash by titration with permanganate, and the values calculated to lead chromate. Of 105 samples examined, 19, or 18 per cent, were colored with lead chromate, the amount of which varied from 0.12 to 1.47 per cent.

Grieb (*107*) gives the ether-soluble matter of a number of insect powders. Nine samples obtained at different times from the same

source gave ether-soluble matter from 7.3 to 12.4 per cent, while 5 samples from another source gave 7 to 12 per cent. Grieb makes a preliminary test with 1 gram of powder, shaking it with 10 cc. of ether in a test tube, and if, after settling, the ether is of a marked green color ("indicating the presence of ground stalks"), the assay is not proceeded with and the sample is rejected.

Jean (*147*), in his analysis of a number of samples of Pyrethrum powder, determined moisture, ash, "acidity," alcohol-ether extract, resinous substances, substances soluble in water, and the iodin absorbed by the water solution after rendering it alkaline by bicarbonate of soda (Table 18). In order to compare these results, Jean prepared a powder from genuine Pyrethrum flowers, called the "type" sample.

TABLE 18.—*Chemical analysis of Pyrethrum powder (Jean)* [1]

Sample	Determination on dry basis						
	Ash	Acidity as H_2SO_4	Alcohol-ether extract	Resinous substances	Soluble in water	Iodin absorbed	Moisture
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
Type	8.90	1.00	24.03	9.30	14.73	3.86	----------
A [2]	7.47	.99	24.94	8.01	16.93	7.65	11.5
B	10.04	1.09	30.47	13.76	16.70	5.22	9.9
C	8.70	.64	21.91	9.41	12.50	3.15	8.0
D	9.02	1.03	24.40	11.14	12.26	5.8	12.5

[1] Jean does not give his methods of analysis.
[2] Contained potassium chromate and sawdust.

Sato (*236*) states that mature flowers of the *Chrysanthemum cinerariæfolium* should be used in preparing insect powder. The most desirable moisture content is 7 to 8 per cent; the ash is 6 to 7 per cent, and always shows the reaction for manganese. The greater the content of ether-soluble matter the greater the value of the powder. If the ether extract is green, leaves and stalks have been mixed with the flowers. The amount of ether extract should not be less than 6 per cent, and the ratio of ether extract to ash should be greater than 1. H. W. and S. C. Gadd (*90*) give methods for detecting turmeric and chrome alum in insect powder. For the determination of ether-soluble material they use Durrant's method. According to them the ash of a genuine powder should be of a light gray color and should not amount to over one-sixteenth of the original.

Vogt (*286*) in 1906 wrote that he invariably found a rich yellow percolate to be characteristic of the finest insect powders. He obtained 7.72 per cent of ether extract in a sample of insect powder ground by himself from closed flowers, and stated that samples of powders guaranteed by first-class houses to be ground from selected closed flowers have yielded from 7.13 to 10.25 per cent ether extract.

Evans Sons, Lescher & Webb (*76*) give the results shown in Table 19 on the ether extract determination on insect powders.

TABLE 19.—*Ether extract determination on insect powders*

Year	Labeled as—	Ether extract
		Per cent
1906	Closed flowers	8 to 9
1906	Open flowers	5 to 6
1906	Stalks	Up to 5
1907	Closed flowers	8.5 to 8.8
1907	Open flowers	5.8 to 6.2
1909	Powder from closed flowers	6.9
1909	do	6.6
1909	Powder from half-open flowers	5.9
1909	Powder from open flowers	5.2

One sample examined in 1909 gave only 4.3 per cent of an extract heavily contaminated with chlorophyll. Microscopical examination of the powder showed the presence of tissue derived from the stalk of the plant. Eight American commercial insect powders examined in 1910 contained from 3 to 4.8 per cent ether-soluble matter, the green color of the extracts indicating admixture of open flowers and stem tissues. A few foreign-ground powders examined in 1906 yielded from 2 to 3 per cent ether extract. The powders reported in Table 19, examined in 1906 and 1907, were of their own grinding.

Southall Brothers and Barclay in 1910 (*268*) reported finding from 7.57 to 8.28 per cent ether extract in authentic samples of insect powder when determined according to Durrant's method. Japanese flowers, mostly open, gave 13.98 per cent "resin" of an orange-brown color. In 1912 (*269*) they found 3.81 per cent of a deep-green extract in a specimen of insect powder said to be ground from stalks. Leubner (*172*), in 1910, found 5.59 per cent of a dirty-green extract in a sample of insect powder. He macerated the sample with an excess of ether for 3 hours, and dried the extract at 100°.

Cæsar and Loretz (*43*), in 1911, gave their method for the determination of ether extract insect powder as follows: Seven grams of the air-dried powder are treated in a 150 cc. flask with 70 grams of ether, macerated 2 hours, the mixture being frequently shaken by hand, then filtered through a 9 cm. filter; 50.5 grams (=5 grams of powder air-dried) of the filtrate are then evaporated in a 9 to 10 cc. porcelain dish over hot water, being careful not to set the dish on the ring of a steam bath, as the solution in that case will creep over the edge. The evaporation is carried to dryness, and the residue brought to constant weight in a desiccator. The extract should have a golden-yellow color and a characteristic, powerful odor, which should not resemble that of chamomile. This method is somewhat different from that given by them in 1898 (*42*), a slight modification of Durrant's test in which the extraction of the sample with ether was continued as long as the solvent took up anything, instead of exhausting with a specified quantity of ether. This method differs from that published by Fromme in 1900 only in the time of maceration, being two hours instead of one.

Cæsar and Loretz (*42*) found that insect powder made from closed flowers yielded from 8 to 9.5 per cent of extract, while open or partly open flowers gave 6.5 to 7.5 per cent. They state that the color of the extractions varies from pure yellow, dark yellow, and brownish yellow to greenish yellow, whereas that prepared from stems is of a

dirty-green color, and the residue amounts to only 5.5 per cent. They state furthermore that the insecticidal properties of insect powder are fully represented in the ether extract, and are not due to any alkaloidal bodies that the powder may contain.

Linke (*173*) obtained the values shown in Table 20 on six authentic powders.

TABLE 20.—*Chemical analysis of authentic insect powders and whole flowers* (*Linke*)

Sample No.	Determination		
	Moisture (loss at 100–105°)	Ether extract (Durrant's method)	Ash
	Per cent	*Per cent*	*Per cent*
Powder:			
1	9.08	4.28	7.04
2	9.64	5.48	7.52
3	7.20	[1] 5.56	7.64
4	6.66	5.22	
5	6.22	6.26	
6	5.24	5.56	6.56
Whole flowers:			
1	10.72		5.88
2	10.36		6.44
3	10.76		6.44

[1] 6.80 per cent when shaken continuously for 2 hours.

In the ash, the presence of iron, manganese, aluminum, calcium, magnesium, sodium, and potassium was shown, but in no instance was chromium found. After evaporating the ethereal solution the residue was dried at 100° C. for one hour before weighing.

Sattler (*238*) reports finding lead chromate in an insect powder, the ash of which was 6.105 per cent. Wiebelitz (*293*), in 1912, found a lower ash content in two samples of insect powder than that usually reported, namely, 4.7 per cent and 5.1 per cent. Siedler (*150*), in 1912, obtained the results given in Table 21 on pure insect powder and on stem powder.

TABLE 21.—*Chemical analysis of pure insect powder and stem powder* (*Siedler*)

Product	Determination		
	Moisture at 110°	Ash on air-dried material	Ether extract
	Per cent	*Per cent*	*Per cent*
Pure insect powder	10.77	6.81 to 7.29	
Powder from best flowers			6.14
Stem powder	8.69	6.39	1.48
Unpowdered, but finely-cut flowers		6.36	

Siedler used ether with a specific gravity of 0.720, and, after completely extracting the sample in the cold, allowed the ether to evaporate spontaneously, and dried the residue for one hour upon the water bath. The color of the extract of the powdered flowers was dirty yellow, that of the stem powder green. With concentrated sulphuric acid there was no characteristic color reaction with either resinous residue. Siedler found the petroleum-ether extract of pure

powdered flowers to be 4.01 per cent, and that of stem powder, 1.01 per cent. The flower extract was a pure yellow, that of the stems a pure green. With concentrated sulphuric acid the petroleum-ether extract from the flowers gave a deep green color, the stem powder a brown. He attempted to obtain a basis for analysis by means of the optical behavior of an alcoholic extract from flower and stem powders, but this extract proved to be optically inactive.

In 1913 Siedler (*257*) commented as follows on the chemical examination of insect powder:

As the best criterion for the value of the insect powder is the determination of the ether extract, which was first made by Thoms. Later this method was modified in this way, that the powder was shaken with ether and an aliquot part of the decanted ether solution allowed to evaporate. This procedure has certain drawbacks. It is not possible to decant a definite quantity of ether solution without causing a difference in weight by evaporation of ether. Further, the ether solution can not be obtained clear except after long standing and it is very difficult to filter clearly. It always deposits fine dust after filtration. Finally, there is generally the feeling that the powder is not completely extracted through simple shaking, that a part of the ether-extractable material escapes determination.

In the same article Siedler gives results for the determination of ash in insect powders (Table 22). All these samples were ground from the best flowers free from stems, and had an average moisture content of 4 per cent. The total ash and the ash insoluble in dilute hydrochloric acid were determined on 2.5 grams of substance.

TABLE 22.—*Determination of ash in insect powder* (*Siedler*)

Sample No.	Total	Insoluble in HCl	Sample No.	Total	Insoluble in HCl
	Per cent	*Per cent*		*Per cent*	*Per cent*
1	8.6	0.1	6	8.0	0.1
2	7.7	.1	7	7.9	.1
3	7.0	.2	8	7.7	.1
4	7.0	.2	9	7.8	.1
5	7.9	.1	10	6.8	.1

Fromme (*86*) reports that the ash content of insect powder is between 6.5 and 9.5 per cent. Later (*87*) he criticizes the method of Trottner, whereby the value of the powder is determined by the number of pollen grains present. Fromme found that some of the most active powders contain fewer pollen grains than other powders prepared largely from stems. The analytical results on seven samples tested are given in Table 23.

TABLE 23.—*Examination of active insect powder* (*Fromme*)

Ash	Ether extract	Color of ether extract	Pollen grains in 1 mg.
Per cent	*Per cent*		
8.21	6.37	Golden yellow	308
7.17	6.00	Greenish yellow	2,415
7.25	6.15	Golden yellow	2,900
8.00	5.85	Greenish yellow	230
8.84	6.27	Golden yellow	3,000
8.00	5.01	Dirty green	710
7.10	3.40	Greenish	294

Windisch (*830*), in 1921, reported the results of analyses of five samples of commercial insect powder sold as "Zacherlin." The total ash content varied from 6.84 to 9.78 per cent, and the sand content (ash insoluble in hydrochloric acid) from 0.27 to 0.88 per cent. Costa (*87*, *88*) has described a method of determining the aqueous extract of insect powder. Calculated on a dry basis, the aqueous extract of closed insect flowers varies from 22 to 25 per cent; that of open flowers from 12 to 14 per cent; and that of stems from 9 to 11 per cent.

SUMMARY OF METHODS

Satisfactory chemical and microscopical methods have been developed for detecting the addition of other species of flowers, curcuma, and Pyrethrum stems to genuine insect powder. These methods, however, do not show accurately the extent of this adulteration, although the quantity of stems can be approximated by comparing under the microscope the unknown mixture with known mixtures of powdered flowers and stems. Of course, the quantity of an inorganic adulterant like lead chromate may be accurately determined by chemical analysis, but such adulteration is now rare. Probably more than 90 per cent of the adulteration of insect powder at the present time is with ground Pyrethrum stems.

A promising microscopic method for examining insect powder is that proposed by Trottner, in which the number of pollen grains in the weighed quantity of sample is estimated. This number, however, varies so much in different powders of equal effectiveness that a rigorous quantitative application of the method is impossible.

Kuraz has done more than anyone else to determine the relative effectiveness of insect flowers and stems. The individual variations in his results, however, lessen the value of his method of testing against flies.

Of the chemical methods proposed the most valuable is the determination of the quantity, and more particularly the color, of the ether extract. The quantity of ether extract in flowers and stems, however, varies markedly, and its determination alone is not sufficient to enable the analyst to establish standards.

No element or compound that can be detected with the present methods of analysis occurs exclusively in either the stem or flower of the Pyrethrum plant.

WRITERS' METHOD

The addition of ground stems of the Pyrethrum plant to the powdered flowers is now the chief form of sophistication. As the quantity of added stem can not be accurately determined by the microscope, although it can be approximated, a chemical method for measuring this form of adulteration has been devised.

The insecticidal ingredients of Pyrethrum flowers are known, but no method for an accurate quantitative determination of them has been devised. It is therefore necessary to rely upon the determination of some essential constituent present in the flowers in a reasonably definite quantity and either absent from other parts of the plant or present in a very different quantity. Most of the constituents of the flowers that can be accurately and readily determined occur also in all other parts of the plant, although in varying quantities. The proportions of nitrogen and phosphorus in the flowers are large as

compared with those in the stems. Upon these two constituents therefore the most stress is laid. Although the active insecticidal principles are soluble in ether and certain other organic solvents, the efficiency of the product can not be measured by the quantity of this ether extract as now determined, because some inactive constituents are also soluble in ether and many of the purest samples show an ether extract content lower than that of some which are seriously adulterated. The color of the ether extract, which should be observed before evaporating off the ether, is of more value than the quantity in determining the purity of the powder. It should be yellow, with no more than a slight tinge of green.

The chemical composition of a number of genuine samples of Pyrethrum flowers and stems was studied. Most of the samples were the *Chrysanthemum cinerariæfolium*, the species now commonly used in the United States for the production of insect powder. The three grades of flowers recognized by the trade, namely, "open," "half-open" ("half-closed"), and "closed," were obtained. Samples of the commercial flowers, consisting of from 1 to 2 pounds of material, were obtained, and the percentage amounts of stems and dirt (including all matter other than flowers or stems) present were first determined. From the commercial samples of Pyrethrum stems the burrs, straw, and other foreign matter were removed and classified as "dirt." In each case 100 grams of the material, selected so as to accurately represent the whole sample, was used, and the separations were carefully made by hand. After separation of the extraneous material the flowers and stems were each ground to a powder for chemical analysis. (Owing to the small quantity of "dirt" and stems in the commercial flowers and to the small quantity of dirt in the stems, it was found that these had little influence on the composition of the stems and flowers. The removal of this extraneous matter was therefore discontinued.)

Methods of Analysis

Moisture.—Two grams of powder was weighed into an aluminum dish provided with a tight-fitting cover, and dried to constant weight in an oven heated by boiling water, *in vacuo*. When cooling in the desiccator and when weighing, the dishes were kept covered, so that the powder could not absorb any moisture from the air. It was found necessary to heat the powder from 8 to 10 hours before constant weight was reached. The loss in weight was reported as moisture.

Nitrogen.—This was determined in 2 grams of powder according to the official Gunning method.[5]

Ash.—Four or five grams of the sample was weighed into a platinum dish and slowly heated in an electric muffle furnace, finally at a dull red heat, until all the carbon was consumed. The residue was reported as ash. In practically all published methods for the determination of ash in insect powder, it is stated that the powder is simply ignited to constant weight. Such a method might give erroneous results, owing to the presence of volatile potassium salts in Pyrethrum, which would be lost; also, ignition in the presence of organic matter would reduce phosphates. By using an electric muffle furnace all the carbon is consumed at a dull red heat; hence, these losses are practically prevented.

[5] Methods of Analysis, A. O. A. C., 2 ed., 1925, p. 8.

Ash insoluble in HCl ("sand").—The residue from the ash determination was transferred to a beaker, and digested with dilute hydrochloric acid (water 100 cc., HCl, sp. gr. 1.19, 15 cc.) for two hours on the steam bath, then filtered and washed, and the residue ignited and weighed. Since the ash in different samples varies, the ash insoluble in hydrochloric acid was calculated back to the original sample.

Phosphorus in the ash.—The filtrate from the last determination was made up to volume in a graduated flask, and phosphoric acid determined in an aliquot according to the official volumetric method (Methods of Analysis, A. O. A. C., 2 ed., p. 3), allowing the solution to stand 30 minutes or longer at about 45°. The phosphorus, reported as P_2O_5, is calculated on the original sample. In many cases the P_2O_5 was determined gravimetrically (Methods of Analysis, A. O. A. C., 2 ed., p. 2).

RESULTS OF ANALYSIS (1911 TO 1917)

TABLE 24.—*Examination of "closed" flowers (Chrysanthemum cinerariæfolium)*

Sample No.	Source [1]	Stems	"Dirt"	Determination [2]				
				Moisture	Nitrogen, N	Ash	Ash insoluble in HCl	P_2O_5
		Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
12321 [3]	Japan	1.00	0.25	4.50	1.58	6.80	0.67	0.715
12325	Europe	3.85	.75	4.65	1.83	7.84	.16	.728
12327	do	3.85	.15	4.40	1.74	7.38	.17	.622
12334 [4]	do	6.25	.30	4.87	1.65	7.29	.15	.604
12335 [3]	Japan	1.55	.15	4.62	1.72	7.09	.48	.709
14920	Europe	1.30		5.10	1.75	6.61	.33	.744
14921	do	2.70		5.70	1.92	7.12	.05	.740
14929	do	6.44	.38	5.51	1.65	6.12	.21	.754
14932 [3]	Japan	.57	.11	5.71	1.64	6.19	.26	.729
14934	Europe	1.68		5.08	1.73	6.24	.11	.679
14938	do	.81		6.15	1.74	6.54	.36	.770
15009	do	3.08		5.94	1.80	7.43	.10	.743
15136 [4]	do	5.05		7.55	1.87	7.69	.17	.815
17389	do	2.20	Trace.		1.69	6.97	.08	.624
17394 [3]	Japan	1.12	Trace.		1.81	6.91	.24	.751
17395	Europe	1.54	Trace.		1.59	7.07	.13	.621
17621 [4]	do	4.27	Trace.		1.85	7.36	.08	.706
17749	do	2.03	Trace.		1.78	7.70	.11	.591
17750	do	2.36	Trace.		1.73	6.86	.13	.659
Minimum		.57	.11	4.40	1.580	6.12	.05	.591
Maximum		6.44	.75	7.55	1.920	7.84	.67	.815
Average		2.72	.30	5.37	1.741	7.01	.21	.700
No. samples examined		19	7	13	19	19	19	19

[1] Practically all of the samples reported in this table were shipped from the port of Trieste, Austria.
[2] The flowers only were analyzed.
[3] Stated to be of Japanese origin and to have been repacked in Austria, but there is no proof of this.
[4] Imported as wild Montenegrin flowers.

TABLE 25.—*Examination of "closed" flowers (Chrysanthemum cinerariæfolium)*

Sample No.	Determination [1]			
	Nitrogen, N	Ash	Ash insoluble in HCl	P_2O_5
	Per cent	*Per cent*	*Per cent*	*Per cent*
Imported from Europe, supposedly of Dalmatian origin:				
12653	1.77	7.66	0.10	
14752	1.55	6.25	.35	
14991	2.00	7.91	.09	
15271 [2]	1.72	6.56	.33	
15344	1.64	6.21	.26	

[1] Results determined on the samples as received, including attached stems and the small amount of dirt present.
[2] Stated to be of Japanese origin.

TABLE 25.—*Examination of "closed" flowers (Chrysanthemum cinerariæfolium)*—Con.

Sample No.	Determination[1]			
	Nitrogen, N	Ash	Ash insoluble in HCl	P_2O_5
Imported from Europe, supposedly of Dalmatian origin—Con.	*Per cent*	*Per cent*	*Per cent*	*Per cent*
16260	1.54	6.29	0.26	....
16261	1.54	6.28	.30	....
17821	1.80	7.94	.13	....
18008	1.83	7.46	.09	....
18152	1.85	8.28	.31	....
18160[2]	1.77	8.22	.39	....
18376	1.95	7.74	.12	....
18548	1.86	7.54	.13	....
18598	2.09	8.06	.14	....
18724	1.82	7.61	.11	....
19055	1.82	8.15	.16	....
19175	1.67	6.72	.28	....
19179	1.71	7.42	.16	....
19284	1.91	7.52	.13	....
19286	1.92	7.73	.11	....
19288	1.89	8.06	.16	....
19924	1.79	8.03	.08	....
20188	1.75	7.08	.08	....
20189	1.60	7.14	.11	....
21208[3]	1.75	6.90	.64	....
21334	1.60	7.30	.15	....
21370	1.78	6.22	.28	....
21919	1.78	7.96	.13	....
22078	1.65	7.53	.21	....
22178	1.87	7.48	.09	....
22179	1.79	6.22	.39	....
22296	1.81	7.35	.18	....
22574	1.71	6.98	.43	....
22695	1.53	6.85	.14	....
22781	1.90	7.98	.86	....
23248	1.72	7.36	.91	....
Minimum	1.53	6.21	.08	....
Maximum	2.09	8.28	.91	....
Average (36 samples)	1.76	7.33	.24	....
Japanese origin:				
17927	1.69	6.36	.35	0.620
21163	1.74	6.27	.25	.758
21186	2.01	6.03	.19	.730
21439	1.84	6.68	.34	.642
21740	1.74	6.63	.41	.693
21840	1.94	6.16	.27	.663
22577	1.84	6.61	.29	.636
22698	1.78	6.99	.38	.697
22944	1.96	6.75	.28	.725
22949	1.66	6.95	.32	....
23247	1.65	6.48	.29	....
23438	1.72	6.67	.31	....
23772	1.61	7.41	.90	.729
23915	1.75	7.18	.42	....
24074	1.82	6.85	.65	....
24078	1.78	6.67	.29	....
24080	1.72	6.92	.68	....
24084	1.76	7.09	.79	....
24108	1.77	7.01	.74	....
24109	1.79	6.88	.47	....
24110	1.80	7.15	.82	....
24114	1.94	7.43	.97	....
24115	1.76	7.70	1.21	....
24116	1.73	7.19	.77	.684
24117	1.82	7.39	.97	.647
24118	1.76	6.96	.60	.624
24119	1.81	7.40	.98	.686
24120	1.81	7.26	.81	....
24121	1.88	7.33	.79	....
24298	1.80	6.81	.31	....
24368	1.74	7.13	.61	....
24625	1.84	8.58	2.03	.662

[1] Results determined on the samples as received, including attached stems and the small amount of dirt present.

[2] Stated to be wild Montenegrin flowers.

[3] Stated to be of Japanese origin.

TABLE 25.—*Examination of "closed" flowers (Chrysanthemum cinerariæfolium)*—Con.

Sample No.	Determination			
	Nitrogen, N	Ash	Ash insoluble in HCl	P_2O_5
Japanese origin—Continued.	*Per cent*	*Per cent*	*Per cent*	*Per cent*
24626	1.84	7.72	1.11	....
24627	1.91	7.46	.89	....
24628	1.87	7.50	.94	....
24629	1.82	7.63	1.05	....
24630	1.81	7.84	1.22	....
24631	1.80	7.72	1.34	....
24632	1.90	8.22	1.83	....
24633	1.70	7.28	1.04	....
24634	1.93	7.26	1.07	....
24635	1.90	8.31	1.77	....
24636	1.91	7.82	1.36	....
24637	1.98	7.68	1.15	....
24638	1.87	7.59	1.05	....
24639	1.84	7.37	.99	....
24640	1.91	7.12	.76	....
24641	1.77	7.22	.94	....
Minimum	1.61	6.03	.19	0.620
Maximum	2.01	8.58	2.03	.758
Average	1.813	7.18	.79	.680
No. samples examined	48	48	48	15

TABLE 26.—*Examination of "half-closed" flowers (Chrysanthemum cinerariæfolium)*

Sample No.[1]	Stems	"Dirt"	Determination [2]				
			Moisture	Nitrogen, N	Ash	Ash insoluble in HCl	P_2O_5
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
12330	3.95	0.25	4.97	1.63	7.34	0.12	0.599
12331	5.05	.35	4.90	1.41	6.88	.45	.530
14926	2.90	....	6.40	1.74	6.97	.19	....
14935	1.16	....	6.44	1.55	....	....	....
17392	1.91	.14	....	1.75	7.60	.47	....
17419	0.62	....	....	1.58	6.14	.34	....
17620	2.28	....	....	1.74	6.88	.41	....
17673	2.22	.81	....	1.69	7.91	.21	....
18549	....	....	....	1.47	6.65	.31	.528
18603	....	....	....	1.48	6.63	.13	....
19056	....	....	....	1.40	7.57	.47	.592
19057	....	....	....	1.47	7.95	.74	....
20726	....	....	....	1.74	7.38	.50	....
21920	....	....	....	1.45	6.72	.15	....
22077	....	....	....	1.50	6.70	.23	....
22079	....	....	....	1.51	7.40	.35	....
22177	....	....	....	1.56	6.55	.10	....
22237	....	....	....	1.52	6.93	.10	....
Minimum	0.62	0.14	4.90	1.40	6.14	0.10	0.528
Maximum	5.05	.81	6.44	1.75	7.95	.74	.599
Average	2.51	.39	5.68	1.56	7.07	.31	.562
No. samples examined	8	4	4	18	17	17	4

[1] All the samples in this table were imported from Europe and are supposedly of Dalmatian origin, except No. 20726, which was stated to be of Japanese origin.
[2] Where the percentage of stems is given, the chemical analysis was made on the flowers only; on the other samples the analysis was made on the material as received.

Table 27.—*Examination of "open" flowers (Chrysanthemum cinerariæfolium)*

Sample No.	Stems	"Dirt"	Determination[1] Moisture	Nitrogen, N	Ash	Ash insoluble in HCl	P_2O_5
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
12320	1.40	0.30	4.81	1.24	5.71	0.10	0.534
12323	1.70	.65	5.33	1.26	6.26	.23	.585
12324	3.10	.20	5.55	1.30	6.55	.17	.528
12329	3.50	.65	5.40	1.31	6.12	.16	.551
12332	2.75	.45	5.65	1.40	7.09	.35	.557
14923	4.51	....	6.13	1.31	6.42	.31	.593
14924	3.61	....	7.70	1.31	5.68	.13	.605
14927	2.45	....	7.41	1.26	5.61	.11	.550
14930	1.93	....	6.35	1.22	4.88	.09	.588
14936	2.87	.30	6.61	1.27	5.15	.09	.598
14939	1.97	....	6.82	1.33	7.06	.65	.618
14945	1.85	....	9.21	1.28	6.10	.28	.537
14961	2.91	....	7.10	1.26	6.15	.28	.586
14962	2.05	....	6.44	1.28	5.76	.17	.615
15008	1.79	....	7.65	1.24	5.68	.10	.588
15209	1.09	....	7.85	1.25	5.56	.08	.593
15270	1.60	....	7.69	1.25	5.97	.18	.507
17385	2.36	Trace	....	1.26	5.93	.26	.541
17388	2.48	do	....	1.23	5.89	.24	.510
17391	2.23	do	....	1.19	6.09	.26	.457
17393	2.14	do	....	1.27	6.63	.41	.453
17396	1.17	do	....	1.22	5.73	.23	.524
17418	1.50	do	....	1.26	6.11	.46	.485
17422	2.38	do	....	1.25	6.16	.31	.486
17623	1.53	do	....	1.25	6.97	.53	.562
17674	3.48	do	....	1.26	6.21	.39	.459
17751	2.28	do	....	1.18	5.28	.19	.518
17929	1.45	do	....	1.36	6.11	.29	.517
Minimum	1.09	0.20	4.81	1.18	4.88	.08	.453
Maximum	4.51	.65	9.21	1.40	7.09	.65	.618
Average	2.29	.43	6.69	1.268	6.03	.25	.544
No. samples examined	28	6	17	28	28	28	28

[1] Flowers only analyzed.

Table 28.—*Examination of "open" flowers (Chrysanthemum cinerariæfolium)* [1]

Sample No.	Stems	Determination. Nitrogen, N	Ash	Ash insoluble in HCl	P_2O_5
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*
12459	3.40	1.25	6.10	0.13	0.537
12460	2.80	1.27	5.94	.08	.490
12652	2.70	1.17	6.29	.19	....
12703	4.00	1.25	6.36	.11	....
12790	1.90	1.27	5.72	.11	.459
12860	4.00	1.37	6.47	.50	.499
14383	....	1.26	6.08	.15	.517
14892	....	1.31	6.89	.47	.513
14977	....	1.23	6.17	.46	.451
15343	....	1.27	6.10	.22	.514
15530	....	1.24	5.76	.31	.462
15542	....	1.24	5.99	.23	....
16377	....	1.20	6.33	.31	.528
16757	....	1.25	6.04	.32	....
16863	....	1.26	5.84	.33	.546
16987	....	1.24	5.68	.24	.506
16998	....	1.30	6.39	.42	.595
17754	4.99	1.26	6.12	.42	.455
17822	1.28	1.23	6.06	.24	.497
17928	1.40	1.30	6.38	.27	.519
18003	2.34	1.23	5.57	.09	.566
18004	1.66	1.24	5.76	.34	.515
18005	.88	1.29	6.13	.08	.546
18006	.96	1.30	6.10	.08	.561
18007	1.30	1.30	6.84	.41	.507
18009	1.89	1.21	5.96	.08	.543
18010	2.26	1.23	5.83	.24	.512
17871	3.07	1.32	5.46	.17	.530
18117	....	1.22	5.69	.25	.460
18118	....	1.25	6.02	.13	.530
18150	....	1.24	6.74	.62	.490
18154	....	1.23	6.49	.49	.528
18377	....	1.25	5.96	.19	.483

[1] The results of analysis are on the material as received. The samples are of European origin.

TABLE 28.—*Examination of "open" flowers (Chrysanthemum cinerariæfolium)*—Con.

Sample No.	Stems	Determination			
		Nitrogen, N	Ash	Ash insoluble in HCl	P_2O_5
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*
18378		1.20	5.65	0.16	0.469
18379		1.34	6.19	.15	.549
18380		1.27	5.85	.13	.503
18381		1.19	5.73	.12	.472
18382		1.31	5.88	.20	.519
18547		1.19	5.22	.14	.515
18597		1.25	5.68	.09	.485
18599		1.28	5.73	.13	.531
18600		1.29	6.32	.28	.514
18601		1.27	5.76	.14	.547
18604		1.23	5.64	.09	.472
18725		1.23	5.38	.16	.541
19178		1.29	6.12	.22	.521
19240		1.24	5.83	.21	.608
19241		1.37	6.02	.22	.592
19285		1.33	5.96	.06	.532
19287		1.27	5.68	.18	.523
19290		1.31	6.28	.21	.595
19291		1.32	6.93	.37	.532
19292		1.38	6.53	.31	.554
19627		1.21	7.53	1.38	.561
19925		1.20	6.08	.25	.511
19926		1.21	5.01	.08	.505
20145		1.21	5.74	.27	.493
20436		1.23	5.64	.21	.592
20437		1.25	5.93	.09	.479
20438		1.31	5.91	.13	.508
20439		1.25	6.40	.37	.495
20440		1.19	6.24	.14	.501
20442		1.30	5.92	.14	.601
20443		1.18	6.19	.12	.559
20444		1.24	7.00	.30	.539
20446		1.38	6.80	.37	.568
21106		1.26	6.45	.26	.612
21184		1.32	5.95	.22	.667
21368		1.33	5.67	.14	.538
21918		1.25	5.36	.13	.576
22424		1.27	5.92	.09	
22694		1.37	7.87	.88	
22696		1.25	6.06	.20	
22697		1.36	7.57	.48	
22925		1.39	6.62	.10	
24302		1.28	6.66	.41	
Minimum	.88	1.17	5.01	.06	.451
Maximum	4.99	1.39	7.87	1.38	.667
Average	2.40	1.267	6.11	.25	.526
No. samples examined	17	76	76	76	66

TABLE 29.—*Examination of insect flower stems (cleaned) (Chrysanthemum cinerariæfolium)* [1]

Sample No.	"Dirt"	Determination				
		Moisture	Nitrogen, N	Ash	Ash insoluble in HCl	P_2O_5
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
12322		4.77	0.842	3.98	0.10	0.144
12326	Trace.	5.20	.695	4.00	.11	.151
12328		5.10	.842	5.00	.38	.200
12333		5.22	.856	4.69	.31	.247
12336		4.72	.863			
14922	4.37	5.34	.820	4.58	.38	.269
14925	8.27	4.51	.800	4.86	.27	.146
14928	1.06	5.79	.575	4.34	.15	.183
14931	.38	5.09	.800	4.63	.59	.169
14937	4.44	5.34	.960	5.44	.38	.256
14946	5.67	6.39	.800	4.29	.30	.236
14960	.96	5.69	.805	5.12	.34	.182
15010	1.35	5.26	.705	4.78	.18	.203
15137	8.89	7.20	.870	5.19	.41	.128
Minimum	.38	4.51	.575	3.98	.10	.128
Maximum	8.89	7.20	.960	5.44	.59	.269
Average	3.93	5.40	.802	4.68	.30	.193
No. samples examined	9	14	14	13	13	13

[1] All samples were imported from Europe.

TABLE 30.—*Examination of insect flower stems (commercial) (Chrysanthemum cinerariæfolium)*

Sample No.	Determination			
	Nitrogen, N	Ash	Ash insoluble in HCl	P_2O_5
Imported from Europe (supposedly of Dalmatian origin):	*Per cent*	*Per cent*	*Per cent*	*Per cent*
17397	0.677	4.83	0.38	0.191
17622	.725	4.06	.21	.212
17748	.674	4.61	.29	.185
17820	.877	4.78	.50	.191
17823	.940	4.26	.09	.145
18161	.779	4.74	.41	.166
18380	.589	4.19	.21	.137
18550	.568	4.11	.26	.150
19289	.772	3.94	.13	.190
20187	.835	5.01	.37	.190
20266	.582	6.03	.33	.117
24190	.877	6.70	.44	.345
24294	.547	3.60	.14	.257
Minimum	.547	3.60	.09	.117
Maximum	.940	6.70	.50	.345
Average	.726	4.68	.29	.190
No. samples examined	13	13	13	13
Japanese origin:				
21369	.610	3.40	.06	.125
22576	1.05	5.62	.34	.383
22936	.863	6.22	.72	.236
23773	.982	5.93	1.69	.338
24295	1.12	5.97	.69	.388
24369	1.10	6.24	1.41	.411
92	.982	5.03	1.10	.326
93	.814	5.58	1.50	.324
94	.954	5.07	.79	.347
95	.948	5.63	1.45	.336
96	.884	4.81	.90	.327
25087	.828	5.02	.80	.374
Minimum	.610	3.40	.06	.125
Maximum	1.120	6.24	1.69	.411
Average	.928	5.38	.95	.326
No. samples examined	12	12	12	12

TABLE 31.—*Nitrogen in stems picked from "open" flowers (Chrysanthemum cinerariæfolium)*

Sample No.	Nitrogen, N	Sample No.	Nitrogen, N
	Per cent		*Per cent*
12332	0.673	17422	0.620
14923	.674	17623	.740
14924	.590	17674	.620
14927	.702	17751	.700
14930	.646	17754	.590
14936	.730	17822	.786
14939	.730	17871	.730
14945	.646	17928	.650
14961	.702	17929	.670
14962	.730	18003	.646
15008	.954	18004	.730
15209	.710	18005	.842
15270	.790	18006	.823
17385	.650	18007	.748
17388	.700	18010	.646
17391	.700		
17393	.670	Minimum	.590
17396	.600	Maximum	.954
17418	.930	Average (34 samples)	.708

TABLE 32.—*Nitrogen in stems picked from "closed" flowers (Chrysanthemum cinerariæfolium)*

Sample No.	Nitrogen, N	Sample No.	Nitrogen, N
	Per cent		*Per cent*
14921	1.60	17749	1.52
14934	1.40	17750	1.52
15009	1.52	17821	1.57
15136	1.54	18008	1.63
17389	1.54		
17394	1.77	Minimum	1.21
17395	1.21	Maximum	1.77
17621	1.54	Average	1.53

It was found that a determination of the nitrogen and of the phosphorus, together with a qualitative ether-extract test (to determine its color), were sufficient to determine the presence and approximate percentage of stems.

In but few of the published analyses of insect powder, where ether extract is given as one of the determinations, is the kind of ether specified. In some methods the powder is simply shaken with ether and filtered, and an aliquot evaporated. Dietze (*70*) has shown that the results do not agree when ether of specific gravity 0.720 and 0.735 is used. Ordinary ether contains some alcohol and water and extracts from insect-powder material other than the active insecticidal constituents. For concordant results, the ether used in making the extractions must always be of the same grade. Anhydrous, alcohol-free ether would seem to be the best solvent.

Pyrethrum flowers usually yield an intensely yellow ether extract, whereas the stems give one of a strong green color. The odor of the extract from the flowers is also characteristic. In the examination of commercial insect powder the presence of a material amount of powdered stems can always be detected by the green color of the ether extract. Its quantitative estimation is, however, of little value in determining the quantity of stems present, and this determination was made in only a few cases, the method employed being as follows: The 2 grams used in the determination of moisture were transferred to an extraction thimble and extracted in a suitable apparatus for 16 hours with ether that had been freshly distilled over metallic sodium. After extraction, the ethereal solution was evaporated to dryness on the steam bath and dried to constant weight at the temperature of boiling water.

The petroleum-ether extract was determined in a few cases by thoroughly extracting 2 grams of the powder in a Soxhlet apparatus with commercial petroleum ether (specific gravity, 0.640; boiling point, 30° to 65° C.), evaporating the extract on the steam bath, and drying for a few minutes in an oven at 100° C.

The petroleum-ether extract of flowers does not differ enough from that of stems to make this determination of value in determining the amount of stems present.

Pentosans were determined in a few of the samples, and it was found that the amounts in the different forms of flowers differed but little from each other and also but little from that in the stems.

The determination of crude fiber is of value in indicating whether or not the powder has been made from open or closed flowers. Pentosans and crude fiber were determined according to the methods of the Association of Official Agricultural Chemists (Methods of Analysis, A. O. A. C., 2 ed., pp. 117, 120).

TABLE 33.—*Determination of ether extract and petroleum-ether extract*

Sample No.[1]	Determination	
	Ether extract	Petroleum-ether extract
"Closed" flowers:	*Per cent*	*Per cent*
14920	----------	4.75
14921	7.45	----------
14929	7.18	5.03
14932	6.85	----------
14934	5.99	3.05
14938	7.34	3.93
15009	6.41	3.80
Minimum	5.99	3.05
Maximum	7.45	5.03
Average	6.87	4.11
No. samples analyzed	6	5
"Open" flowers:		
14923	5.48	3.64
14924	5.73	3.98
14927	6.28	4.45
14930	----------	4.70
14936	6.00	----------
14939	----------	3.53
14945	6.60	5.05
14961	5.35	3.50
"Open" flowers—Continued.	*Per cent*	*Per cent*
14962	5.93	3.60
15008	5.09	----------
Minimum	5.09	3.50
Maximum	6.60	5.05
Average	5.81	4.06
No. samples analyzed	8	8
Stems:		
14922	3.15	1.95
14925	3.35	2.08
14928	2.88	1.93
14931	2.34	1.53
14937	3.98	----------
14946	3.43	1.75
14960	----------	2.78
15010	3.40	1.78
Minimum	2.34	1.53
Maximum	3.98	2.78
Average	3.22	1.97
No. samples analyzed	7	7

[1] All samples were obtained from Europe.

TABLE 34.—*Determination of pentosans*

Sample No.[1]	Pentosans
"Closed" flowers:	*Per cent*
12321	17.75
12325	16.06
12327	16.18
12334	16.53
12335	16.80
Minimum	16.06
Maximum	17.75
Average (5 samples)	16.66
"Open" flowers:	
12320	21.42
12323	21.07
12324	21.72
12329	21.34
"Open" flowers—Continued.	*Per cent*
12332	19.98
Minimum	19.98
Maximum	21.72
Average (5 samples)	21.11
Stems:	
12322	18.40
12326	18.78
12328	17.54
12333	18.40
12336	17.95
Minimum	17.54
Maximum	18.78
Average (5 samples)	18.21

[1] All samples were obtained from Europe.

TABLE 35.—*Determination of crude fiber*

Sample No. [1]	Crude fiber
	Per cent
"Closed" flowers:	
12334	22.89
14921	18.65
14929	20.63
14932	19.80
14934	20.62
14938	20.03
17389	20.94
17394 [2]	22.73
17395	21.96
17621	20.39
17749	21.61
17750	19.45
17927 [3]	23.83
21163 [3]	21.97
21186 [3]	21.47
21439 [3]	24.70
21740 [3]	21.71
21840 [3]	24.03
22577 [3]	23.91
22698 [3]	22.47
22944 [3]	21.46
23772 [3]	24.10
24116 [3]	23.54
24117 [3]	22.86
24118 [3]	23.77
24119 [3]	23.78
24625 [3]	23.03
Minimum	18.65
Maximum	24.70
Average (27 samples)	22.09
"Open" flowers:	
12320	35.12
12323	30.13
12324	31.55
12329	29.79
12860	27.20
14936	33.11
14945	27.75
14961	29.48
14962	30.28
14977	31.18
15008	30.03
17393	29.17
17751	34.70
17754	29.48
17929	32.80
18547	30.13
19292	30.27
20145	32.23
20436	33.87
20437	27.63
"Open" flowers—Continued.	
20438	32.54
20439	31.17
20440	30.11
20442	32.13
20443	31.99
20446	28.82
21184	29.96
21918	35.85
Minimum	27.20
Maximum	35.85
Average (28 samples)	31.02
Dalmatian stems:	
14925	34.55
14928	36.45
14931	33.54
14937	39.05
14946	35.43
17397	38.82
17622	38.92
17748	38.27
17820	39.71
17823	38.23
18380	38.61
18550	39.13
20187	38.10
24190	33.60
24294	42.98
Minimum	33.54
Maximum	42.98
Average (15 samples)	37.69
Japanese stems:	
21369	39.07
22576	46.19
22936	32.39
23773	46.12
24295	44.46
24369	42.99
24392	46.24
24393	47.45
24394	48.05
24395	47.31
24396	48.79
25087	43.49
Minimum	32.39
Maximum	48.79
Average (12 samples)	44.38
Average of stems	40.66

[1] Unless otherwise indicated, all samples were obtained from Europe.
[2] Source unknown.
[3] Obtained from Japan.

The ash content is highest in closed flowers, next highest in open flowers, and lowest in the stems. A higher percentage of the ash of stems is insoluble in hydrochloric acid than in the case of the flowers, although calculated back to the original material the difference is slight. It is only when the ash and the ash insoluble in acid are markedly above the normal for flowers that much importance is attached to these determinations.

A striking thing in the figures for nitrogen and for phosphoric acid is that they run fairly constant for the same material (*Chrysanthemum cinerariæfolium*), no matter what its source. This uniformity in the amount of certain ingredients present in Pyrethrum flowers and stems

aids the analyst to determine from the analysis of an unknown commercial insect powder whether or not it is adulterated with powdered Pyrethrum stems.

By the color of the ether extract and by a determination of the nitrogen, phosphoric acid, and crude fiber, together with a microscopical examination, the presence of powdered stems in insect powder can be shown qualitatively and an approximate determination of the amount of stems can be made.

Table 36 summarizes all the results for nitrogen, phosphorus pentoxid, crude fiber, ash, ash insoluble in hydrochloric acid, pentosans, ether extract, moisture, and extraneous material, for stems and the three commercial grades of flowers, of both Dalmatian and Japanese origin.

TABLE 36.—*Summary of examination of insect flowers and stems*

Product		Nitrogen, N	P_2O_5	Crude fiber	Ash	Ash insoluble in HCl	Pentosans	Ether extract	Petroleum-ether extract	Moisture	Stems	"Dirt"
		Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
Japanese "closed" flowers	Min	1.61	0.620	21.46	6.03	0.19	------	------	------	------	------	------
	Max	2.01	.758	24.70	8.58	2.03	------	------	------	------	------	------
	Ave	1.813	.680	23.11	7.18	.79	------	------	------	------	------	------
No. samples examined		48	15	15	48	48	------	------	------	------	------	------
Dalmatian "closed" flowers	Min	1.53	0.591	18.65	6.12	0.05	16.06	5.99	3.05	4.40	0.57	0.11
	Max	2.09	.815	22.89	8.28	.91	17.75	7.45	5.03	7.55	6.44	.75
	Ave	1.759	.700	20.81	7.22	.23	16.66	6.87	4.11	5.37	2.72	.30
No. samples examined		55	19	12	55	55	5	6	5	13	19	7
All "closed" flowers (Japanese and Dalmatian)	Min	1.53	0.591	18.65	6.03	0.05	------	------	------	------	------	------
	Max	2.09	.815	24.70	8.58	2.03	------	------	------	------	------	------
	Ave	1.784	.691	22.09	7.20	.49	------	------	------	------	------	------
No. samples examined		103	34	27	103	103	------	------	------	------	------	------
"Half-closed" flowers	Min	1.40	0.528	------	6.14	0.12	------	------	------	4.90	0.62	0.14
	Max	1.75	.599	------	7.95	.74	------	------	------	6.44	5.05	.39
	Ave	1.566	.562	------	7.07	.31	------	------	------	5.68	2.51	.81
No. samples examined		18	4	------	17	17	------	------	------	4	8	4
"Open" flowers	Min	1.17	0.451	27.20	4.88	0.06	19.98	5.09	3.50	4.81	0.88	0.10
	Max	1.40	.667	35.85	7.87	1.38	21.72	6.60	4.70	9.21	4.99	.90
	Ave	1.267	.532	31.02	6.09	.26	21.11	5.81	4.06	6.69	2.33	.47
No. samples examined		104	94	28	104	104	5	8	8	17	45	9
Japanese stems	Min	0.610	0.125	32.39	3.40	0.06	------	------	------	------	------	------
	Max	1.12	.411	48.79	6.24	1.69	------	------	------	------	------	------
	Ave	.928	.326	44.38	5.38	.95	------	------	------	------	------	------
No. samples examined		12	12	12	12	12	------	------	------	------	------	------
Dalmatian stems	Min	0.547	0.117	33.54	3.60	0.09	17.54	2.34	1.53	4.51	------	0.38
	Max	.960	.345	42.98	6.70	.59	18.78	3.98	2.78	7.20	------	8.89
	Ave	.733	.192	37.69	4.68	.29	18.21	3.22	1.97	5.40	------	3.93
No. samples examined		6	26	15	26	26	5	7	7	14	------	9
All stems (Japanese and Dalmatian)	Min	0.547	0.117	32.39	3.40	0.06	------	------	------	------	------	------
	Max	1.12	.411	48.79	6.70	1.69	------	------	------	------	------	------
	Ave	.765	.234	40.66	4.90	.50	------	------	------	------	------	------
No. samples examined		18	38	27	38	38	------	------	------	------	------	------

In estimating the quantity of stems in an unknown sample, its nitrogen and phosphorus pentoxid contents are compared with the

average values for these determinations in samples of flowers and stems.[6]

The formula for making the calculations is as follows:

$$X=\frac{100\ (a-c)}{a-b}$$

in which X = percentage of stems in sample.

a = average percentage of nitrogen or P_2O_5 in flowers.

b = average percentage of nitrogen or P_2O_5 in stems.

c = nitrogen or P_2O_5 in sample.

The value of a differs as to whether the mixture is made with open flowers and stems or with closed flowers and stems.

In the case of open flowers and stems:

(1) Based on nitrogen values—

$a=1.267$

$b=0.765$

$$X=\frac{100\ (1.267-c)}{1.267-0.765}=200\ (1.267-c)$$

(2) Based on P_2O_5—

$a=0.532$

$b=0.234$

$$X=\frac{100\ (0.532-c)}{0.532-0.234}=336\ (0.532-c)=\frac{100\ (0.532-c)}{3}$$

In mixtures of closed flowers and stems:

(1) Based on nitrogen values—

$a=1.784$

$b=0.765$

$$X=\frac{100\ (1.784-c)}{1.784-0.765}=98\ (1.784-c)$$

(2) Based on P_2O_5—

$a=0.691$

$b=0.234$

$$X=\frac{100\ (0.691-c)}{0.691-0.234}=220\ (0.691-c)$$

In connection with other observations, the crude fiber determination is of value in indicating whether open or closed flowers have been used in the mixture. In most cases these mixtures are made from open flowers, which are cheaper than closed, and stems, though sometimes closed flowers are employed. The question then arises, how can the analyst tell whether open or closed flowers have been used and which values to apply in the formula.

This may be determined in the following ways:

1. Examination by a microscopist will show whether open or closed flowers have been used. The presence of a large quantity

[6] In obtaining these averages the results of analyses of all cleaned and uncleaned flowers and stems, and also of commercial samples of flowers (with a small part of the stem left attached when harvested), were included. Theoretically the averages should have been based either on cleaned flowers, without attached stems, and on cleaned stems, or on commercial flowers as marketed (with the dirt and short stems still present), and on commercial stems containing the normal percentage of foreign matter. However, the quantity of stems and foreign matter in commercial flowers and the quantity of foreign matter in stems are so small that the error introduced in this way is negligible.

of pollen and the absence of fruit tissues indicate closed flowers; and, conversely, the absence of much pollen and the presence of a large amount of fruit tissues indicate open flowers. These tissues are very characteristic and can not be mistaken (Pl. IV).

2. Mixtures of flowers and stems are made up on the basis of lowest cost. By following the market prices on closed and open flowers and stems, the analyst can usually tell which has been used in preparing a mixture of flowers and stems.

3. From the intensity of the green color of the ether extract, after a little experience, the amount of stems present can be told roughly.

4. The crude fiber determination, taken in connection with the intensity of the green color of the ether extract, general appearance of the powder, and odor, serves as a good indicator as to whether or not the mixture is composed of open flowers and stems or closed flowers and stems.

By a combination of all of these tests, but more particularly by microscopical examination, it is possible to tell whether closed or open flowers have been used in a mixture, and, by the use of the formula, to determine the approximate percentage amount of flowers and stems present.

In Table 37 are given the nitrogen and phosphorus pentoxid contents of 10 known mixtures of open flowers with stems, and the quantity of stems calculated from each of these values, using the formulas just given.

TABLE 37.—*Nitrogen and phosphorus pentoxid content of known mixtures of open flowers and stems and quantity of stems present*

Sample No.	Stems	Nitrogen, N	P_2O_5	Stems calculated from—		Mean	Error
				Nitrogen, N	P_2O_5		
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
1	0	1.280	0.530	3	1	2	+ 2
2	0	1.235	.523	6	3	5	+ 5
3	0	1.250	.510	3	7	5	+ 5
4	10	1.185	.459	16	24	20	+10
5	10	1.170	.482	19	17	18	+ 8
6	20	1.140	.411	25	40	33	+13
7	20	1.170	.447	19	28	24	+ 4
8	60	1.035	.275	46	86	66	+ 6
9	60	.919	.300	70	77	74	+14
10	80	.947	.206	64	100	82	+ 2

Tables 38 and 39 give the results of the analysis of commercial insect powders. Those reported in Table 38 are on the samples unadulterated with stems, foreign plant material, or coloring matter, while those in Table 39 are on samples adulterated with ground Pyrethrum stems, sand, chromates, or other materials.

TABLE 38.—*Analysis of commercial insect powders unadulterated with Pyrethum stems, foreign plant material, or coloring matter*

Sample No.	Made in—	Nitrogen, N	Ash	Ash insoluble in HCl	Sample No.	Made in—	Nitrogen, N	Ash	Ash insoluble in HCl
		Per ct.	*Per. ct.*	*Per ct.*			*Per ct.*	*Per ct.*	*Per ct.*
11157	United States	1.64	8.92	0.84	21371	Japan	1.78	7.09	.65
11292	do	1.29	6.73	.22	21941	United States	1.73	8.06	1.02
12033	do	1.55	8.07	.98	21969	do	1.31	6.54	.28
12034	do	1.56	9.01	1.33	22180	Japan	1.66	7.49	1.71
12212	England	1.52	7.67	.43	22637	United States	1.28	6.69	.53
12217	United States	1.56	7.67	.95	22641	do	1.45	8.08	.97
12273	do	1.65	9.93	--------	22701	do	1.73	8.10	1.19
12613	England	1.63	7.54	.17	22756	"Imported"	1.33	10.00	[1] 2.47
12654	United States	1.26	7.22	.66	22764	United States	1.69	7.35	.84
12657	do	1.35	8.57	[1] 2.22	22804	do	1.47	10.09	1.49
12660	do	1.25	6.49	.24	22894	do	1.41	8.71	1.24
12920	do	1.54	7.09	.33	22895	do	1.66	9.07	.51
13202	do	1.75	7.08	.75	22968	do	1.30	6.81	.51
13603	do	1.40	7.70	.99	22972	do	1.67	8.90	.59
13649	do	1.53	7.25	.89	23071	do	1.33	8.04	1.24
13695	Austria	1.26	6.29	.33	23246	do	1.65	8.47	.34
13768[2]	United States	1.60	7.37	1.47	23330	do	1.28	7.09	.67
13860	do	1.61	7.58	.95	23353	do	1.64	7.03	.78
13921	do	1.54	7.15	.74	23403	do	1.42	7.36	.76
13923	do	1.57	6.93	.68	23491	do	1.83	7.26	.64
13985	do	1.23	6.05	.27	23561	do	2.01	7.05	.70
14197	do	1.14	6.11	.39	23647	do	1.85	7.10	.77
14370	do	1.35	6.90	.64	23648	do	1.74	6.89	1.02
14331	do	1.72	7.03	.13	23655	do	1.65	6.53	.58
14445	do	1.26	5.77	.26	23727	do	1.48	8.19	1.11
14523	do	1.24	8.14	.97	23918	Japan	1.43	9.67	4.32
14730	do	1.66	7.57	.75	23990	do	1.86	7.47	.91
14992[2]	do	1.62	7.12	1.00	23949	United States	1.77	6.85	.90
15153	Austria	1.76	7.83	.36	23969	do	1.70	7.06	.70
15224	Italy	1.49	7.00	.31	24077	do	1.89	6.56	.45
15272	United States	1.41	8.48	1.69	24083	do	1.84	7.19	.77
15366	do	1.21	7.09	.96	24112	do	1.88	7.61	1.21
15730	Austria	1.29	6.23	.39	24123	do	1.76	7.40	1.00
15850	United States	1.54	7.02	.33	24124	do	1.77	7.47	.94
16018	Japan	1.66	7.19	.67	24125	do	1.74	7.47	.92
16019	do	1.72	6.91	.49	24126	do	1.81	7.53	1.03
16499	England	1.54	8.34	[1] 2.00	24127	do	1.65	6.84	.82
16546	United States	1.48	8.09	1.01	24189	Japan	1.80	6.90	.55
16611	do	1.30	7.75	1.64	24276	United States	1.81	7.75	1.07
16796	Japan	1.71	6.69	.44	24277	do	1.73	7.48	1.04
17459	do	1.66	6.39	.46	24278	do	1.85	8.23	1.45
17619	United States	1.82	8.64	.15	24279	do	1.77	7.71	1.09
17624	do	1.64	7.74	.93	24280	do	1.68	7.50	.99
17671	Germany	1.64	7.43	.09	24281	do	1.73	7.72	1.31
17672	do	1.53	7.49	.09	24282	do	1.79	8.58	1.82
18022	England	1.82	8.23	1.26	24283	do	1.70	7.42	1.06
18149	United States	1.23	7.00	.12	24284	do	1.79	7.37	.86
18151	do	1.80	8.72	.29	24285	do	1.76	6.97	.64
18156	do	1.46	8.20	.83	24286	do	1.87	8.15	1.33
18159	do	1.95	8.36	.36	24287	do	1.80	8.19	1.64
18383	do	1.27	7.78	1.09	24301	do	1.80	7.23	.68
18622	do	1.48	6.91	.56	24303	do	1.74	8.76	1.90
18719	do	1.52	7.04	.31	24307	do	1.35	7.98	.61
19187	Japan	1.72	7.38	.91	24314	do	1.67	7.69	.51
19273	England	1.61	7.83	.95	24315	do	1.68	7.71	.53
19283	Australia	1.28	6.54	.18	24316	do	1.70	7.70	.51
19320	United States	1.24	6.74	.66	24317	do	1.59	7.58	.62
19643	do	1.20	7.73	[1] 2.42	24318	do	1.55	7.57	.64
19660	do	1.34	6.70	.34	24319	do	1.59	7.42	.62
19661	do	1.56	7.46	.43	24320	do	1.65	7.66	.54
19671	do	1.56	7.42	.81	24321	do	1.54	7.46	.64
19825	do	1.15	7.25	1.59	24322	do	1.63	7.92	1.11
19833	do	1.28	6.99	.78	24323	do	1.59	7.88	1.52
20097	do	1.44	7.67	1.22	24324	do	1.49	7.38	.98
20224	do	1.24	6.49	.51	24325	do	1.51	8.06	1.67
20225	do	1.08	7.76	2.42	24326	do	1.43	8.04	1.55
20226	do	1.13	6.03	.33	24327	do	1.43	7.81	1.46
20250	do	1.15	6.15	.27	24328	do	1.45	7.83	1.60
20373	do	1.26	7.48	1.15	24329	do	1.51	7.65	1.53
20456[2]	do	1.30	5.47	.69	24330	do	1.45	8.02	1.78
20462	do	1.20	6.79	.80	24331	do	1.49	7.93	1.87
20472	do	1.26	7.11	.62	24332	do	1.59	9.26	[1] 3.61
20658	do	1.18	6.80	.70	24333	do	1.52	7.17	1.05
20771	do	1.21	6.98	.70	24334	do	1.65	7.62	1.37
21043	do	1.60	7.29	1.51	24335	do	1.62	7.89	1.41
21148	do	1.22	6.73	.56	24336	do	1.43	7.57	1.77

[1] Contains sand.

[2] *C. cinerariæfolium* grown in California.

Table 38.—*Analysis of commercial insect powders unadulterated with Pyrethrum stems, foreign plant material, or coloring matter*—Continued

Sample No.	Made in—	Nitrogen, N	Ash	Ash insoluble in HCl	Sample No.	Made in—	Nitrogen, N	Ash	Ash insoluble in HCl
		Per. ct.	*Per ct.*	*Per ct.*			*Per ct.*	*Per ct.*	*Per ct.*
24337	United States	1.48	7.68	1.74	24646	United States	1.77	7.43	1.05
24338	do	1.45	6.67	.91	24647	do	1.77	7.64	1.27
24339	do	1.54	7.84	1.29	24648	do	1.73	7.56	1.14
24340	do	1.48	7.87	1.90	24649	do	1.77	7.55	1.13
24341	do	1.39	7.34	1.73	24650	do	1.77	7.42	1.12
24342	do	1.41	7.91	2.15	24651	do	1.80	7.93	1.46
24343	do	1.51	7.78	1.53	24652	do	1.80	7.59	1.10
24344	do	1.45	7.62	1.96	24653	do	1.52	6.59	.81
24345	do	1.79	7.89	.58	24708	Europe	1.34	7.77	.72
24346	do	1.73	7.59	.46	24736[2]	United States	1.59	7.35	1.08
24347	do	1.63	7.03	.40	24779	Europe	1.20	7.07	1.27
24348	do	1.67	7.24	.35	24798	United States	1.56	7.80	1.22
24349	do	1.71	7.32	.33	24812	Japan	1.59	8.63	[1] 2.99
24350	do	1.62	7.41	.99	24820[2]	do	1.66	6.86	.66
24351	do	1.62	7.46	.70	25062	United States	1.68	7.54	1.18
24352	do	1.63	7.41	.87	25069	do	1.26	6.75	.20
24353	do	1.65	8.01	1.04	25070	do	1.68	8.01	.29
24354	do	1.56	7.53	.91	26188[2]	do	1.32	5.53	.44
24355	do	1.70	7.71	.43	33035[2]	do	1.56	6.46	.68
24356	do	1.65	7.84	.73					
24426	do	1.51	7.46	1.49		Minimum	1.08	5.47	.09
24437	do	1.73	6.87	.69		Maximum	2.01	10.09	4.32
24643	do	1.76	7.44	1.04		Average	1.55	7.50	.95
24644	do	1.76	7.57	1.15		Number samples			
24645	do	1.79	7.81	1.29		examined	196	196	195

[1] Contains sand. [2] *C. cinerariæfolium* grown in California.

Table 39.—*Analysis of adulterated commercial insect powders*

Sample No.	Made in—	Nitrogen, N	Ash	Ash insoluble in HCl	Color of ether extract	Adulterants
		Per ct.	*Per ct.*	*Per ct.*		
11301	United States	0.895	10.26	3.32	Strong green	Stems and sand.
11819	do	1.555	7.72	1.28	do	Stems.
11831	do	.884	6.28	.69	do	Do.
12031	do	.891	6.76	1.83	do	Stems and sand.
12073	do	1.080	7.17	1.88	do	Do.
12303	do	1.060	9.86	3.56	Dirty pale yellow	Do.
12466	do	1.122	12.70	5.50	Green	Do.
12658	do	1.135	7.77	1.90	do	Do.
13238	do	1.120	7.06	.57	do	Stems.
13286	do	.910	5.43	.07	do	Do.
13287	do	1.090	7.81	.16	do	Do.
13311	do	1.120	6.24	.57	Greenish	Do.
13823	Italy	1.370	8.71		Slightly greenish yellow.	Lead chromate (2.82 per cent).
13824	Austria	.990	7.31		Strong green	Stems and 2.27 per cent lead chromate.
13857	United States	1.180	7.60	.86	Greenish yellow	Stems and unidentified bark.
13925	do	1.140	9.78	2.10	do	Stems and sand.
14039	do	.950	6.37	.69	Strong green	Stems.
14105	do	1.145	9.60	2.26	do	Stems and sand.
14362	do	.947	6.77	1.54	do	Stems.
14550	do	.919	6.31	.67	do	Do.
14854	do	1.120	6.12	.55	Greenish yellow	Do.
15393	Austria	1.210	7.30	.55	Green	Stems and 1.48 per cent lead chromate.
16920	do	.820	23.43		do	Stems, calcium carbonate, and 2.07 per cent barium chromate.

TABLE 39.—*Analysis of adulterated commercial insect powders*—Continued

Sample No.	Made in—	Nitrogen, N	Ash	Ash insoluble in HCl	Color of ether extract	Adulterants
		Per ct.	*Per ct.*	*Per ct.*		
17384	United States	0.954	7.14	0.85	Green	All stems.
17420	do	.856	5.70	.90	do	Do.
17998	do	.947	5.66	.56	do	Stems.
18153	do	.849	7.62	1.14	do	All stems.
19145	do	.807	6.36	.61	Strong green	Stems.
19846	do	1.030	6.69	1.42	do	Do.
20765	do	1.185	7.18	1.13	Greenish yellow	Do.
20786	do	.887	6.06	.37	Strong green	Do.
20787	do	1.060	7.19	.74	Green	Do.
20793	do	.954	6.16	.61	do	Do.
20940	do	.772	4.96	.59	Strong green	Do.
21115	do	1.145	10.12	4.31	Greenish yellow	Stems and sand.
22215	do	1.015	6.09	.40	Dirty green	Potassium chromate and stems.
23161	do	.786	5.27	.66	Strong green	All stems.
23369	do	1.040	6.57	.80	Green	Stems.
23479	do	1.165	6.75	.83	do	Do.
23547	do	1.090	7.05	.70	do	Do.
24030	do	1.050	8.68	3.18	do	Stems and sand.
24768	do	1.120	7.00	.76	do	Stems.
24769	do	.898	6.38	.69	Strong green	Do.
25046	do	1.170	6.10	.82	Green	Do.

TABLE 40.—*Analysis of commercial powders containing stems, amounts declared, made from "open" flowers and stems*

Sample No.	Nitrogen, N	P_2O_5	Stems stated to be present	Stems calculated from—		Mean	Error
				Nitrogen, N	P_2O_5		
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
12031	0.891	0.289	75	75	81	78	+3
12073	1.080	.402	50	37	43	40	−10
12658	1.135	.385	50	26	49	38	−12
14039	.950		50	63			+13
14362	.947	.284	75	64	83	74	−1
19846	1.030		50	47			−3
20786	.887	.316	70	76	72	74	+4
20787	1.060	.390	40	41	47	44	+4
20793	.954		50	62		62	+12
23161	.786		100	96			−4
23369	1.040		50	45			−5
23547	1.090		50	35			−15
24030	1.050		50	43			−7

From Table 40 it is evident that the amount of stems in commercial insect powder determined by the proposed method of calculation from the values for nitrogen and phosphorus pentoxid agrees within an average of less than 10 per cent of the amount declared to be present.

In some cases the ash and acid-insoluble ash contents in insect powders have been found to be higher than they should be, based on those determinations made on samples of known purity and on samples of flowers and stems. Some jobbers have claimed that the high ash content of their product was due to the addition by the manufacturer of siliceous material during the grinding or to the introduction of mineral matter during the grinding as a result of abrasion from the mill. In the case of stone mills there would be

some wearing of the stones and a small quantity of material would be introduced into the powder in this way. It can readily be seen, however, that if this were sufficient to increase the ash content appreciably, say, 1 per cent, there would soon be nothing left of the mill.

In order to determine whether there was any marked difference in the composition of the powder from that of the flowers from which it was made, a number of samples of closed Japanese flowers were collected immediately before entering the mill and samples of the ground product as it came from the mill. Owing to the great capacity of the milling and sieving machinery, the powder from any particular sample of flowers could not be identified, but when collected over a period of a working day an average of all samples collected should be representative of the material. In Table 41 are given the analyses of these samples, which represent a run of about 60 bales (25,000 pounds) of closed Japanese flowers. Table 42 contains the results of analyses of Japanese stems before and after grinding.

TABLE 41.—*Analysis of Japanese closed flowers before and after grinding*

Flowers			Powder		
Nitrogen, N	Ash	Ash insoluble in HCl	Nitrogen, N	Ash	Ash insoluble in HCl
Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
1.96	6.75	0.28	1.735	6.77	0.53
1.66	6.95	.32	1.705	7.15	.74
1.82	6.85	.65	1.89	6.56	.45
1.78	6.67	.29	1.84	7.19	.77
1.72	6.92	.68	1.88	7.61	1.21
1.76	7.09	.79	1.76	7.40	1.00
1.77	7.01	.74	1.77	7.47	.94
1.79	6.88	.47	1.74	7.47	.92
1.80	7.15	.82	1.81	7.53	1.03
1.94	7.43	.97	1.65	6.84	.82
1.76	7.70	1.21	1.81	7.75	1.07
1.73	7.19	.77	1.73	7.48	1.04
1.82	7.39	.97	1.85	8.23	1.45
1.76	6.96	.60	1.77	7.71	1.09
1.81	7.40	.98	1.68	7.50	.99
1.81	7.26	.81	1.73	7.72	1.31
1.88	7.33	.79	1.79	8.58	1.82
1.84	8.58	2.03	1.70	7.42	1.06
1.84	7.72	1.11	1.79	7.37	.86
1.91	7.46	.89	1.76	8.97	.64
1.87	7.50	.94	1.87	8.15	1.33
1.82	7.63	1.05	1.80	8.19	1.64
1.81	7.84	1.22	1.76	7.44	1.04
1.80	7.72	1.34	1.76	7.57	1.15
1.90	8.22	1.83	1.79	7.81	1.29
1.70	7.28	1.04	1.77	7.43	1.05
1.93	7.26	1.07	1.77	7.64	1.27
1.90	8.31	1.77	1.73	7.56	1.14
1.91	7.82	1.36	1.77	7.55	1.13
1.98	7.68	1.15	1.77	7.42	1.12
1.87	7.59	1.05	1.80	7.93	1.46
1.84	7.37	.99	1.80	7.59	1.10
1.91	7.12	.76	1.52	6.59	.81
1.77	7.22	.94			
[1] 1.83	[1] 7.39	[1] .96	[1] 1.77	[1] 7.56	[1] 1.10

[1] Average

TABLE 42.—*Analysis of Japanese Pyrethrum stems*[1] *before and after grinding*

Unground stems			Powdered stems		
Nitrogen, N	Ash	Ash insol. in HCl	Nitrogen, N	Ash	Ash insol. in HCl
Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
0.982	5.03	1.10	0.706	4.29	0.85
.814	5.58	1.50	.765	4.54	1.02
.854	5.07	.79	1.015	6.26	1.94
.948	5.63	1.45	.877	5.50	1.57
.884	4.81	.90	.891	5.75	1.66
			.842	5.03	1.15
			.954	5.86	1.54
			.891	5.71	1.69
			.912	5.71	1.72
			.926	5.88	1.80
			.758	4.75	1.15
			.961	6.03	1.84
			.940	5.81	1.81
			.891	5.67	1.67
			.863	5.07	1.28
			.870	5.57	1.55
[2] .896	[2] 5.22	[2] 1.15	[2] .879	[2] 5.46	[2] 1.52

[1] These stems originally contained much "dirt" which was removed by sieving, 16 per cent being thus thrown away. The figures reported are on the stems after removal of all dirt. The discarded sievings analyzed:

Nitrogen, N	Ash	Ash insoluble in HCl
Per cent	*Per cent*	*Per cent*
1.93	21.62	13.99
1.93	23.23	15.67
1.82	23.64	16.19

[2] Average.

The average results for nitrogen, total ash, and ash insoluble in hydrochloric acid in the flowers before and after grinding agree closely, showing that no appreciable change in composition is brought about by the process of grinding.

In the case of stems the figures for total ash and acid-insoluble ash are a little higher in the ground product. This is no doubt due in part to the sampling, as it is difficult, in the case of the unground stems, to obtain a uniform sample, owing to the nature of the material and to the way in which it is packed.

Practically all the insect flowers imported into this country are the *Chrysanthemum cinerariæfolium*, but after the beginning of the World War a few shipments of flowers of *C. roseum* were received from Russia, through Marseilles or Italian ports. The results of analysis of these samples are given in Table 43. A comparison of these results with the corresponding figures for the various grades of flowers and stems of *C. cinerariæfolium* shows that the ash and nitrogen contents of *C. roseum* are higher and the ether extract and pentosans are lower than those of either the flowers or stems of the *C. cinerariæfolium*. The values for crude fiber and phosphorus

pentoxid of *C. roseum* correspond closely to those of the closed flowers of *C. cinerariæfolium.* The powder prepared from the flowers of *C. roseum* is much darker and has a different odor than the ordinary insect powder. The odor of an insect powder and a microscopical examination should serve as a means to determine whether or not the product has been prepared from *C. roseum* or *C. cinerariæfolium.*

TABLE 43.—*Analysis of insect flowers (Chrysanthemum roseum)*

Sample No.	Nitrogen, N	P_2O_5	Ether extract	Pento-sans	Crude fiber	Ash	Ash insoluble in HCl
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
17879	2.08					8.75	0.60
18011	2.08					8.45	.35
20487	2.26	0.714	2.83	13.78	20.44	11.09	1.11
21737	1.95	.691	2.74	13.21	20.04	10.89	1.45
22076	2.04	.738	2.73	13.42	19.31	11.90	1.51
22295	1.97					12.62	3.42
22575	2.08	.640			18.46	9.15	.45
22693	2.00	.581			17.80	9.88	1.26
Minimum	1.95	.581	2.73	13.21	17.80	8.45	.35
Maximum	2.26	.738	2.83	13.78	20.44	12.62	3.42
Average	2.08	.673	2.77	13.47	19.21	10.34	1.27

Samples of *C. leucanthemum* (generally called "field daisy" or "oxeye daisy" in this country), which have been largely used in the past, and to some extent at the present time (*S26*), as an adulterant of insect powder, have been analyzed in the Insecticide and Fungicide Laboratory (Table 44).

TABLE 44.—*Analysis of Chrysanthemum leucanthemum*

Sample No.	Description	Nitrogen, N	Ash	Ash insoluble in HCl	P_2O_5	Ether extract	Pento-sans	Crude fiber
		Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
14933 [1]	Flowers only	1.82	10.02	0.92	0.684	3.18	14.34	20.14
23020 [2]	do	2.23	10.45	.15				
23020	Stems only	1.45	10.38	.15				

[1] Sent in as "false" insect flowers.
[2] Collected near Markham, Va.

RESULTS OF ANALYSIS (1918 TO 1924)

From 1918 to 1924, inclusive, nearly 1,100 samples of Pyrethrum flowers, of European and Japanese origin, Pyrethrum stems, *Pyrethrum roseum* flowers, and insect powders, both pure and adulterated, were examined. The results of these analyses are reported in Tables 45 and 46. During this period most of the insect flowers imported were labeled "Pyrethrum flowers" or "insect flowers." In reporting the analyses no attempt has been made to separate the samples examined into grades, as "closed," "half-closed," or "open."

TABLE 45.—*Analysis of Pyrethrum flowers and stems (1918 to 1924, inclusive)*

Product	Origin	Number of samples	Composition: Nitrogen, N		Composition: Total ash	Composition: Ash insoluble in HCl
				Per cent	*Per cent*	*Per cent*
Pyrethrum flowers (*C. cinerariæfolium*).	Europe	288	Minimum	1.19	4.34	Trace.
			Maximum	1.97	9.42	1.78
			Average	1.53	6.39	.24
Do	Japan	265	Minimum	1.15	4.55	Trace.
			Maximum	2.27	8.31	2.10
			Average	1.76	6.32	.61
Pyrethrum flowers (*C. roseum*)	France	3		2.08	9.37	.99
				2.07	9.98	1.28
				2.23	9.13	.90
Pyrethrum stems	Europe	28	Minimum	.58	3.30	Trace.
			Maximum	.84	6.63	0.41
			Average	.72	4.84	.21
Do	Japan	3		.94	6.20	.92
				1.56	8.00	2.01
				1.36	7.63	2.65

TABLE 46.—*Analysis of commercial insect powder (1918 to 1924, inclusive)*

Product	Number of samples	Composition: Nitrogen, N		Composition: Total ash	Composition: Ash insoluble in HCl
			Per cent	*Per cent*	*Per cent*
Unadulterated powder	332	Minimum	1.04	5.00	Trace.
		Maximum	2.07	8.77	2.00
		Average	1.59	6.97	.91
Adulterated powder	158	Minimum	.62	3.90	Trace.
		Maximum	2.02	13.10	8.28
		Average	1.43	7.93	2.00

Of the 553 shipments of whole flowers (*C. cinerariæfolium*) examined from 1918 to 1924, inclusive (Table 45), daisy flowers (*C. leucanthemum*) were found in only 8 cases and never in excess of 1 per cent. In 5 samples a few flowers of the genus Anthemis were found (a fraction of 1 per cent). Acid-insoluble ash in excess of 2 per cent was found in only 1 case and in excess of 1½ per cent in only 6 cases.

Of the 490 shipments of commercial insect powder examined from 1918 to 1924, inclusive (Table 46), 158, or more than 32 per cent, were adulterated. Powdered daisy flowers were present in 41 samples from a trace to more than 25 per cent. Thirty-one samples contained too high a proportion of powdered insect flower stems, eight consisting entirely of powdered stems. (Samples labeled as containing stems are not included in the table.) Powdered turmeric, from a trace to more than 10 per cent, was found in 15 samples. Seventy-nine samples contained ash insoluble in hydrochloric acid in excess of 2 per cent, the maximum being 8.3 per cent. One sample contained more than 25 per cent of powdered flowers of an unidentified species; another contained a little cornstarch; and a third contained a small proportion of powdered eucalyptus leaf tissues.

Samples have not been obtained from every importation of insect flowers nor from every interstate shipment of insect powder, but it is

reasonable to suppose that those examined are fairly representative. It is evident that a great many of the insect powders on the market have not been made from the flowers as imported and that adulteration is being extensively practiced.

ACTIVE PRINCIPLE OF INSECT POWDER

PREVIOUS INVESTIGATIONS

The earliest recorded investigation on the active insecticidal constituents of insect powder is that of Ragazzini (*67*), who used the powder from *C. cinerariæfolium* and concluded that its narcotic action was due not to any one substance but to a mixture of substances.

In 1863, Heller and Kletzinsky (*114*) were reported to have found that genuine Persian insect powder contained no narcotic or alkaloidal plant poisons or poisonous mineral substances, but only ethereal oil and santonin, as the active insecticidal constituents. Hanamann (*114*) concluded that Pyrethrum flowers contain no poisonous alkaloid or santonin, but mostly indifferent substances, and that only the ethereal oil in concentrated form can be harmful. He stated that genuine insect powder can have no harmful action on the human organism.

Rother (*234*), in 1876, recorded the results of tests on Pyrethrum, but did not indicate the species used. He proved the absence of alkaloids and stated that the active principle is a glucoside (*persicin*), which can be split up into glucose and an inert body (*persiretin*). He also isolated a yellowish, bitter resin (*persicein*). Later, Rother (*235*) stated that *persicin* is not a glucoside and that the glucose reaction is due to glucose or a gum preexisting in the powder.

Jousset de Bellesme (*63*), in 1876, stated that the toxic principle of insect powder is entirely extracted by alcohol. He isolated the essential oil of Pyrethrum, and proved its harmlessness on insects. Submitting Pyrethrum to the treatment for extracting an alkaloid gave a crystallized principle "which enjoyed to a high degree the toxic properties of the plant." His statements are not accompanied by experimental proof.

Semenoff (*253*), in 1877, obtained flowers of Pyrethrum, presumably *P. roseum* or *carneum*, from Caucasia. Dried at 25° C., the flowers were as active against insects as commercial insect powder. An essential oil obtained by steam distillation proved to be inactive. Distillation in an alkaline solution gave a very small quantity of an alkaloid. By macerating 1 pound of the flowers with 5 pounds of ether for five days and evaporating the extract, a mixture of resin, oil, wax, and acids was obtained. On steam distillation it yielded an ethereal oil that killed insects.

Hager (*111*), in 1878, reported that the insecticidal activity of the flowers of *P. carneum* and *roseum* was due to two substances. One was a body similar to trimethylamin, which was found in the flowers combined with an acid; the other was a resin found in the pollen grains. Hager obtained some of the first substance in combination with hydrochloric acid, added to it some potassium hydroxid solution, and noticed that flies held over the mixture exhibited convulsive movements. The resin when dusted on insects acted very

energetically. Solutions of the powder made with water or with dilute alcohol were found to be entirely inactive.

Dal Sie (*60*), in 1879, claimed to have proven the presence of a free, easily volatilized acid which was found in the ethereal, alcoholic, or aqueous extracts of the powder. From the ether extract of the flowers he obtained not only a crystallizable acid, but also an aromatic smelling acid of oily consistency at ordinary temperature. From the alcoholic extract he obtained a resinous matter resembling a glucoside, probably the same as that found by Rother, since under the influence of dilute sulphuric acid it split up into sugar and another product. According to Dal Sie, since the fumes which result from the incomplete combustion of insect powder exhibit the toxicity of the original powder, the toxic principle must be volatile without decomposition, and the free volatile acid is, therefore, the most active constituent of Pyrethrum.

Textor (*272*), 1881, as the result of his tests, stated that "the active principle of Persian insect powder is, in nature, a soft resin." He pulverized (80-mesh) 1½ ounces of whole flowers (species not indicated), and percolated the powder with benzine for six hours, using 6 fluid ounces. The benzine solution was evaporated, water was added, and the product was again evaporated to remove any volatile oil. The residue was treated with dilute acid and filtered. No alkaloid could be detected in the filtrate by phosphomolybdic acid, Mayer's reagent, or a solution of iodin in potassium iodid. The benzine extract, the benzine extract in alcohol precipitated by acid, and the benzine extract in caustic potash and precipitated by acid, were all poisonous to flies. The benzine extract in alcohol was acid to litmus. A test for essential oil was made by allowing the powder to stand in dilute salt solution for 24 hours and then distilling, but none was obtained.

Hirschsohn (*133*), in 1890, found that alcohol, ether, chloroform, or benzine would dissolve the active principle of insect powder. The extract obtained with these solvents, when added to an inactive powder, e. g., powdered chamomile, produced a powder as active as the original insect powder before extraction. From the work of Hirschsohn, the active principle would appear to be nonvolatile, as he found samples of powder which had stood for five years in paper boxes still as active as fresh powder. Heated at 120° F. insect powder lost its odor, but was as active as ever against flies. To see if the active principle was acid in nature, he treated powder with alcoholic ammonia, also with alcoholic potash, but after drying in the air the powder was as active as ever. Placed for 24 hours in water, the water became acid, but the powder after drying was still active.

Gillette (*96*), in 1889, arrived at the same conclusions as to the absence of toxic substances in the volatile oil of Pyrethrum. He made practical tests upon insects, operating as follows:

> Two or three grams of the powder were first put in a test tube and the dust allowed to settle for a few minutes. Then a loose cotton plug was pushed halfway down in each, upon which were placed the insects, and the tubes were then tightly corked. Dipterons, ichneumons, chalcids, cynipids, and aphids were subjected to this treatment and allowed to remain for different lengths of time, varying from 1 or 2 to 40 hours, without showing any signs of being affected by the volatile oil.

To see if the toxic principle of Pyrethrum would come off at a higher temperature, Gillette made further tests:

> Two cynipids were then similarly inclosed in a tube with fresh powder and the lower end of the tube was held for 10 minutes in a dish of boiling water, the upper

end of the tube being kept cool with a wet cloth. The insects were allowed to remain for a few minutes after the boiling, but showed no signs of being affected by the treatment.

Another tube was then prepared in which were confined other cynipids in a manner similar to the preceding, and the tube was held over the flame of an alcohol lamp and constantly shaken until the powder was entirely browned, care being taken not to burn it. The insects were unhurt by this treatment.

Then a quantity of powder was put in a tube and held in the flame of a lamp until the tube was filled with fumes. These fumes were poured over into another tube in which was an ichneumon, on which they took immediate effect and death ensued in a very few minutes.

Next, three ichneumons were inclosed in separate tubes. One was shaken up with a small quantity of fresh Pyrethrum powder, one with a small quantity of the browned powder spoken of in the second preceding experiment, and one in a small quantity of the partially burned powder spoken of in the preceding experiment. In each case the insect was killed, the fresh and the browned articles acting alike, and the partially burned powder acting somewhat slower.

Three grams of the powder were then put in about 90 cc. of water in a retort and boiled for one-half hour, the vapor being caught and condensed in a receiver. Soon after boiling began a slight odor was noticed escaping about the mouth of the receiver which the neck of the retort did not quite fill. A cynipid was entangled in a bit of cotton and crowded into the opening, where it remained for 12 minutes without being affected.

About one-half of the liquid was sent over in the above experiment, and the distillate was clear, colorless, and almost tasteless and odorless, there being a slight taste resembling steeped hay. The distillate and the residue were used to compare their insecticidal properties with one another and with fresh powder extracted in cold water for 24 hours. The results of these experiments were that the distillate used pure killed but a very small percentage of the lice treated and that the residue was as efficient as the same quantity of powder extracted in cold water.

Gillette found that ether extracted all the insecticidal principle from Pyrethrum, as the extracted powder when dusted upon insects had no effect. The greater part of the ether extract would dissolve readily in water, and this water solution affected insects exactly as if they had been treated with Pyrethrum.

Zuco (*296*), in 1889, made an ethereal extract of the flowers of *Chrysanthemum cinerariæfolium*. After repeated treatment of this with aqueous and alcoholic potash a substance was left which formed yellow crystals and had a melting point of 70° to 100° F. After repeated recrystallization from cold ether a paraffin, $C_{17}H_{36}$, of melting point 64°, was obtained. This dissolved freely in ether, benzene, chloroform, and moderately hot alcohol, but was almost insoluble in cold alcohol. The portion remaining in cold ether was recrystallized until the melting point was above 150°. Pure cholesterin was prepared from either the acetyl or benzoyl derivative of this substance by the action of alcoholic potash. The cholesterin thus obtained had a melting point of 183°; the melting point of its acetyl derivative was 223°, and that of the benzoyl derivative, 246°. It dissolved freely in ether, benzene, and chloroform, and sparingly in hot alcohol, and gave all the reactions for cholesterin.

In 1890, Zuco (*297*, *298*) obtained a glucoside and also an alkaloid from the flowers of *Chrysanthemum cinerariæfolium*. He described the glucoside as crystalline, but not enough was obtained for proper investigation. Later, Zuco (*299*) described the method he used in obtaining the alkaloid, called chrysanthemine, from Pyrethrum flowers. Ten kilograms of flowers was boiled in distilled water for two or three hours and filtered through cloth. The residue was pressed and treated again in the same manner. The extracts were evaporated down to 30 liters, treated with neutral lead acetate and basic lead acetate, neutralized with soda, and filtered, the excess of lead being re-

moved by hydrogen sulphid. After filtration, the liquid was concentrated to about 2 liters, boiled for some time with dilute sulphuric acid, filtered again, and boiled until no more resinous matters were formed. The liquid was then decolorized with animal black, and an excess of the double iodid of potassium and bismuth added. This precipitated a heavy bright red crystalline powder. From this the alkaloid was obtained. According to Zuco this is a colorless sirup soluble in water and in alkalis and in ethyl and methyl alcohol, but not in ether, chloroform, or benzene. The base is described as being optically inactive and physiologically innocuous. The work of Zuco thus shows that the active insecticidal agent of Pyrethrum flowers is not alkaloidal in nature.

Eymard (*77*), in 1890, distilled a mixture of insect powder with 3 parts of water. The distillate was slightly opaline, held in suspension a small quantity of "essence," and presented a strong and characteristic odor of Pyrethrum flowers. Tested upon ants and other insects, however, the distillate was found to have no effect, from which Eymard concludes that the essential oil is not the active insecticidal agent of insect powder.

An ether extract was made, deep yellow and strongly odorous. It gave up nothing to water, but was completely soluble in 95 per cent alcohol and in alkalis. From alkaline solution it was precipitated by acid. From 110 grams of insect powder Eymard obtained 5.6 grams of ether extract. This was dissolved in alcohol and treated with silver nitrate, which precipitated the fatty acids as silver salts, while the silver resinates remained in solution. The ether extract was in this manner shown to consist of 3.8 grams fatty bodies and 1.8 grams resinous matter. Insects placed upon a piece of paper impregnated with this resinous matter manifested extreme agitation and died in about 5 minutes. After exhaustion with ether, the insect powder was successively extracted with 95 per cent alcohol, cold distilled water, and boiling distilled water, but no toxic substances were obtained. No alkaloids were detected. Eymard concludes that the toxic principle of Pyrethrum is found in the part soluble in ether, and more especially in the resin. However, it is his opinion that several factors unite in the rôle of insecticide, as he found that the isolated active principle worked less actively than the original powder.

Schlagdenhauffen and Reeb (*245*), in 1890, distilled 250 grams of the flowers of Pyrethrum in a current of steam, collecting 750 grams of distillate. After filtering through a wet filter, the distillate was extracted with ether and the ethereal solution was separated, filtered, and evaporated at a temperature not exceeding 30° C. A few drops of a green oil toxic to insects were thus obtained. No alkaloid was present in the oil. The aqueous part was acid, but had no injurious effect upon insects. The authors obtained an acid which was toxic to insects, by exhausting with alcohol acidulated with hydrochloric acid, drying, exhausting with ether, shaking the ethereal solution with ammoniacal water, evaporating to dryness, again taking up in water, and filtering. The filtrate contains the ammonium salt of the toxic acid. This acid was also obtained by extracting with alcohol, neutralizing exactly with a solution of potassium hydroxid, evaporating gently to dryness, taking up in water, filtering, and treating the filtrate with a solution of tartaric acid, and extracting the liberated acid with ether. This toxic acid

the authors call pyrethrotoxic acid. Injected into guinea pigs, this acid produced its action in two perfectly distinct stages. In the first, an excitation was produced, being more or less pronounced according to the proportion of matter administered; in the second stage, on the contrary, a complete prostration was produced, accompanied always with paralysis of the lower extremities.

Thoms (*275*), 1890, extracted 10 kilograms of best "closed" Dalmatian flowers with 55° petroleum ether for seven days. Evaporated *in vacuo*, he obtained 230 grams of a greenish yellow extract which had the characteristic odor of insect powder. This extract contained many crystals and was of a waxy consistency. By solution in alcohol and again evaporating, a yellow wax, melting point 54° C., was obtained. By pouring the alcoholic solution into water and setting in the direct sunlight, a white wax, of melting point 56.5°, was obtained. After separation of the waxy bodies from the solution a sugar (dextrose) was shown to be present. Altogether, Thoms succeeded in isolating from insect powder: (1) An essential oil, (2) a volatile acid, (3) a wax, (4) a nonvolatile, potassium permanganate reducing, balsamlike acid, (5) chlorophyll, (6) colophonic acid, (7) tannic acid, (8) a body with alkaloidal properties, (9) a body with glucosidal properties, and (10) sugar. Thoms found the petroleum-ether extract and the essential oil to possess toxic properties, while the nonvolatile acid, the glucoside, and the other substances were harmless to insects.

De Boisse (*64*), 1895, claimed that the active principle of *Pyrethrum cinerariæfolium* is a yellow resin soluble in sulphuric ether, insoluble in water, and very slightly soluble in alcohol, carbon disulphid, and fatty bodies. Alkalis decompose it rapidly. The flowering heads and leaves contain a good deal of this resin; the lower parts of the stems, very little. De Boisse gives no experimental proof for these statements.

Durrant (*73*), in 1897, stated: "The toxic properties of insect powder are due to (1) a volatile oil amounting to 0.5 per cent in picked specimens of closed flowers and much less in open flowers; (2) a soft acid resinous body which is the principle source of the toxic effect. It is found to the amount of 4.8 per cent in selected closed flowers, less than 4 per cent in half-open flowers, and still less in flowers that are fully open; the whole plant apart from the flowers contains mere traces of resin."

Gerard (*93*), 1898, stated that the active principle of Pyrethrum consists of an oleo-resin and an essential oil. These are found principally in the bracts and around the ovaries of the flowers, comparatively little being present in the corollas.

Sato (*236, 237*), as a result of work done in 1905–1907, reports the isolation of a light-yellow, odorless, transparent, sirupy resin from Pyrethrum flowers, which he calls pyretol. At first this is tasteless, but later has a benumbing effect. It is insoluble in water and dilute acids, and soluble in alcohol, ether, and petroleum ether. It is soluble in hot alkaline solutions, but when precipitated from such solutions by acids the resin becomes completely inactive.

Fujitani (*89*), in 1909, conducted an elaborate research on the flowers of *Chrysanthemum cinerariæfolium*, using flowers cultivated in the provinces of Ki-i and Mikawa, Japan. The flowers were ground and the powder soaked for one week in 95 per cent alcohol at room temperature. The alcohol was distilled off, leaving an extract of a

greenish-brown color and a characteristic odor of insect powder, and quite toxic to insects. The yield was about 10 per cent. The alcohol extract was shaken up with water until entirely free from water-soluble substances. The residue was then dissolved in ether, the part insoluble in ether being treated with potassium hydroxid solution, and this then shaken out with ether. The deep-green ethereal solution was freed of chlorophyll, etc., by shaking with 10 per cent caustic-potash solution until the dark green turned to a yellow brown and the alkaline solution was entirely colorless. The ether solution was then shaken out once with dilute sulphuric acid, then with water, and finally the ether was distilled off. This left a clear, yellow-brown mass which gave a neutral reaction and had a sharp and bitter taste and the characteristic odor of insect powder. The yield of this material was 1.4 per cent of the original material. For further purification the substance was dissolved in a little ether mixed with a large amount of petroleum ether, filtered, warmed with the animal charcoal, and again filtered, and the petroleum ether then evaporated. A yellow sirupy mass was left, which had only a feeble odor, and a taste bitter at first and then intensely sharp. It gave a neutral reaction, was soluble in alcohol, ether, etc., but was insoluble in water, acids, and alkalis. It contained no nitrogen. This substance, which seems to be an ester, Fujitani calls pyrethron, and it is, according to him, the active insecticidal principle of Pyrethrum. Pyrethron decomposes even on standing, yielding pyrethrol, which appears to have the formula $C_{21} H_{34} O$. Tested upon different animals, pyrethron showed an action similar to that of veratrine. Fish and insects were very susceptible but protozoa very tolerant. On warm-blooded animals it caused epileptiform convulsions, increased blood pressure, and increased breathing movements.

Reeb (*214*), 1909, criticized Fujitani's work, arguing that treatment of the alcoholic extract with such powerful reagents as 10 per cent solution of potassium hydroxid and sulphuric acid might change bodies dissolved in the alcohol. Therefore it is not certain that the final product obtained by Fujitani, although toxic, is the real active preexisting principle. Reeb extracted Dalmatian insect powder with petroleum ether (specific gravity, 0.670). The petrolic liquids were filtered and evaporated, leaving a soft residue which represented 3.5 per cent of the powder employed. This was treated with successive quantities of hot alcohol in the presence of a little animal charcoal. The alcoholic solutions were filtered and allowed to stand about a month, at the end of which time a resin (melting point, 125° C.) had deposited. This resin Reeb calls pyrethresine. From the alcoholic solution Reeb separates pyrethrotoxic acid by evaporating to dryness, taking up in acetone, adding barium carbonate, evaporating to dryness, taking up in water, decomposing with sulphuric acid, and shaking out with ether, which upon evaporation leaves the pyrethrotoxic acid. From this work, therefore, Reeb verifies the work of himself and Schlagdenhauffen. According to Reeb, the toxic principle of insect powder is an acid called pyrethrotoxic acid, which preexists in the free state in the flowers of Pyrethrum.

In 1912 Yoshimura and Trier (*295*) published the results of their work upon the closed flowers of *C. cinerariæfolium* in search of betains. From 1 kilogram of air-dried powder they obtained 0.2 gram cholin and 0.8 gram stachydrin, both calculated as hydro-

chlorids. They made no tests as to the insecticidal action of these substances, but they are probably inert.

Siedler (*258*), 1915, distilled 30 kilograms of half-closed flowers with dry steam, extracted the distillate with ether, and thus obtained a salvelike mass of strong characteristic odor. The yield was 20.212 grams, or 0.067 per cent of the original material. Spread on unglazed porcelain, all was absorbed but 0.789 gram. By fractional recrystallization from aqueous alcohol two bodies were obtained from this unabsorbed portion: (1) Fine needles, melting point 54–56° C.; and (2) fine plates, melting point 58–60°. Recrystallized from absolute alcohol this substance had a melting point of 62°. Elementary analysis of (1) showed it to correspond closely to the formula $C_{14}H_{30}$. Analysis of (2) showed a formula approximating palmitic acid. The material absorbed by the porous plate was extracted with ether, and this concentrated solution mixed with 2 parts ether and 1 part alcohol. By extraction with sodium bisulphite solution, 0.049 gram of material was obtained of an aromatic odor, but without effect upon insects. By extraction with 2 per cent potassium hydroxid solution, 3.156 grams of material was isolated. No test for phenol could be obtained. An acid of butyric odor was obtained, but it was inert toward insects. Other tests, such as distillation of the material under reduced pressure, are recorded by Siedler, but nothing possessing insecticidal action could be isolated.

Evidently, then, a number of investigations on the nature of the active insecticidal principle of Pyrethrum had been carried out before 1916, but the results vary greatly. Table 47 gives a brief summary of these results.

TABLE 47.—*Summary of investigations on active insecticidal principle of Pyrethrum, 1854 to 1915*

Year	Investigator	Bibliography reference	Species of Pyrethrum used	Active principle considered to be—
1854	Ragazzini (de Visiani)	67	*Cinerariæfolium*	A mixture of substances, nature not determined.
1863	Heller & Kletzinsky (Hanamann).	114	*Roseum* (?)	An essential oil and santonin.
1863	Hanamann	114	do	An essential oil, "persicin."
1876	Rother	234, 235	(?)	
1876	De Bellesme	63	(?)	A "crystallized principle."
1877	Semenoff	253	*Roseum* (?)	An essential oil.
1878	Hager	111	*Roseum* and *Carneum* (?).	A resin and an amin.
1879	Dal Sie	60	(?)	A free volatile acid.
1881	Textor	272	(?)	A soft resin.
1889	Gillette	96	(?)	Soluble in ether.
1889–1894	Zuco	296, 297, 298, 299	*Cinerariæfolium*	Isolated a paraffin, a phytosterol, an alkaloid, and a glucoside, but all were inert.
1890	Hirschsohn	133	*Roseum* (?)	Not acid and not volatile.
1890	Eymard	77	(?)	Principally a resin.
1890	Schlagdenhauffen and Reeb.	245	*Cinerariæfolium*	"Pyrethrotoxic acid."
1890	Thoms	275	do	An essential oil.
1895	De Boisse	64	do	A resin.
1897	Durrant	73	do	A resin and volatile oil.
1898	Gerard	93	do	An oleoresin and volatile oil.
1905–1907	Sato	236, 237	do	A sirupy resin, "pyretol."
1909	Fujitani	89	do	An ester, "pyrethron."
1909	Reeb	214	do	"Pyrethrotoxic acid."
1912	Yoshimura and Trier	295	do	Cholin and stachydrin (inert).
1915	Siedler	258	do	Nothing definite.

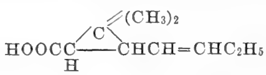
In 1918 Yamamoto (*S31*) obtained a preparation of the active principles of *C. cinerariæfolium* of Japanese origin in a state of purity probably surpassing anything previously reported. He extracted the finely powdered flowers with ether and removed the acid constituents from the partially evaporated extract by 2 per cent caustic soda. The ether was then evaporated and the residue treated with 90 per cent alcohol. This treatment removed a waxy substance. The alcohol was then evaporated in vacuum, and the resulting yellow sirup obtained was taken up in light petroleum ether. Evaporation of the solvent left a light-colored oil, which corresponded to about 0.8 per cent of the powder. This material contained no nitrogen and was soluble in organic solvents but insoluble in water. It consisted largely of an ester, and its iodin value showed that it contained a highly unsaturated compound. Upon treatment with alcoholic potash at room temperature it lost its insecticidal power. Its toxic character was also reduced by heating and long exposure to the air. The preparation, which could not be further purified, is probably practically the same as Fujitani's "Pyrethron." It was highly toxic to flies but had no effect on dogs when introduced into the stomach.

Upon saponification with alcoholic potash solution in the cold, evaporating the alcohol, diluting with water, and extraction with ether, the material gave a mass of crystals and an oily substance. Most of the crystalline material consisted of an alcohol with a melting point of 199° C. and the formula $C_{21}H_{34}O$. Its acetyl compound melted at 222–230°. Several other alcohols were isolated, but Yamamoto believes that they are impure and consist largely of the higher melting compound. The alcohol was not toxic to insects.

The products of saponification of the pyrethron gave an acid fraction in a 50 per cent yield. This acid mixture was esterified with alcohol and hydrochloric acid and the ester was separated by distillation into several fractions. From the lower boiling fractions (77–115°, 1 mm.) a liquid acid ("pyrethronic acid"), having the formula $C_{10}H_{18}O_2$ and one double bond, was obtained. From the higher boiling ester fraction (113–179° C., 1 mm.) a solid acid, identified as palmitic acid, was obtained.

Later work by Yamamoto (*S32*) established the formula $C_{10}H_{16}O_2$ for pyrethronic acid. Reducing the acid with hydrogen in the presence of platinum black gave the saturated dihydropyrethronic acid, represented by the formula $C_{10}H_{18}O_2$. Several salts and derivatives of this acid were prepared and analyzed.

Dihydroxypyrethronic acid was prepared by partial oxidation of pyrethronic acid with permanganate. This compound was further oxidized by potassium dichromate to a dibasic acid, which was shown to be identical with transcaronic acid. The same dibasic acid was obtained by oxidation of pyrethronic acid with ozone. Yamamoto believed that he had identified propionic aldehyde as a second oxidation product and deduced the following formula for pyrethronic acid:



When pyrethronic acid was reesterified with the crude material containing the alcohol component from the saponification of pyrethron, the insecticidal properties of the product were regenerated.

The crystalline alcohol pyrethrol first obtained by Fujitani (*S9*) and described by Yamamoto (*S31*) was not toxic, nor could a toxic product be prepared by combining it with pyrethronic acid. The results of Yamamoto's work indicated that pyrethronic acid is one of the components of the active principle of insect powder.

More recently Yamamoto (*S33*) has described his work on the products of dry distillation of *C. cinerariæfolium*. The material was distilled at a temperature of 260° to 280° C. About one liter of distillate was obtained from 3,500 grams of the powdered flowers. The distillate gave an acid reaction and from it were isolated acetic and propionic acids, acetic and propionic aldehydes, methyl, trimethyl, butyl, and amyl amines, pyridine, and a higher homologue of pyridine whose picrate melted at 84°. These acids and bases seem to be present in the combined form in the flowers.

A study was made of the insecticidal properties of homologous acids, esters, and bases. Of the common fatty acids, formic was the most toxic. Butyl amine showed the greatest toxicity of the monoalkyl amines. In general, the tertiary amines, especially tertiary amyl amine, were the most effective. The insecticidal power increased with the molecular weight among the amines having boiling points below 80°.

Abbott (*S1*), Chevalier (*S3*), Chevalier and Dantony (*S4*), Chevalier and Mercier (*S5*), Costa (*S7, S8*), Gattefossé (*S15*), Juillet (*S17, S18, S19, S20*), and Zeigler (*S34*) have also studied the active constituents of insect powder.

EXPERIMENTAL WORK

Work Done in 1917 [7]

Preliminary tests.—The coarsely powdered flowers, subjected to steam distillation, yielded 0.28 per cent of a fragrant oil. This had an odor somewhat like that of rosemary oil and like the characteristic Pyrethrum odor. The quantity obtained was too small to permit a chemical examination. Tested against flies, this oil had only slight repelling properties, and did not show the characteristic effects of Pyrethrum powder. A steam distillation of the flowers in the presence of sodium hydroxid yielded only 0.15 per cent of an oil which had a disagreeable odor. Careful tests for alkaloids in the distillate gave negative results. A steam distillation of the flowers in the presence of a little sulphuric acid yielded 0.16 per cent of an oil which lacked the characteristic odor of Pyrethrum. Tested against flies, the oils from the alkaline and acid steam distillations had even less effect than that obtained in a straight steam distillation. These experiments indicate that the substance, or substances, in Pyrethrum which cause its characteristic effect upon insects are not removed by steam distillation in neutral, alkaline, or acid solutions.

After being subjected to steam distillation in neutral solution, the flowers were dried and tested upon roaches. They were as active as before treatment, showing that treatment with steam does not decompose the insecticidal principle.

To test the action of dilute acid and alkaline solutions upon insect flowers, 50 grams of the coarsely powdered material was

[7] Conducted by R. C. Roark.

treated with 500 cc. of solution for 24 hours, stirred occasionally, then filtered off, and dried at room temperature. The residual powder, after treatment with 1 per cent hydrochloric acid (HCl), 1 per cent acetic acid (CH_3COOH), 1 per cent ammonium hydroxid (NH_4OH), or distilled water, was apparently as active as ever (tested against roaches), but after extraction with 1 per cent potassium hydroxid (KOH) the powder was entirely inert. These tests show that the active principle is soluble in dilute potassium hydroxid or else is rendered inert by it, but is insoluble in, or unaffected by water, dilute acids, or dilute ammonium hydroxid.

Heated at 107° C. for 17 days, the powder took on a markedly reddish color. All characteristic odor disappeared after about three days' heating. At the end of the 17-day period the powder was tested upon roaches and found to be entirely inert. Heated in a vacuum oven at the temperature of boiling water for 14 hours, the powder completely lost its characteristic odor, but was still active against roaches. This again indicates that the essential oil does not contain the characteristically acting insecticidal principle.

Different portions of the powder were dried over sulphuric acid and solid sodium hydroxid in desiccators *in vacuo* for 12 days. At the end of this time the powders had lost a great portion of their odor but were still active against roaches.

The following tests were made on 300 grams of powder to see what amount of extract different solvents would remove when used one after the other in a Soxhlet extraction apparatus. The succession used was petroleum ether, chloroform, acetone, and 95 per cent alcohol:

Solvent	Extract
	Per cent
Petroleum ether	3.4
Chloroform	5.0
Acetone	6.2
Alcohol	9.0
Total	23.6

Fifty-gram portions of coarsely powdered material (made from partially open flowers) were then completely extracted in a Soxhlet extraction apparatus by various organic solvents. The object of these extractions was to determine what solvents would remove the active principle and at the same time extract as little as possible of the other substances. In nearly all cases a quantity of resinous material separated from solution after the extraction had been running for some time. This resinous material, which would not go into solution, even when treated with large quantities of the solvent, apparently resulted from the polymerization of unstable terpenelike substances extracted by the solvent, a polymerization probably induced by the heat of the boiling solvent.

After extraction the material was tested on roaches, ants, and other insects by the entomologists of the Bureau of Entomology, of this department, at the testing laboratory in Vienna, Va. In all cases the extracted material was entirely inert when tested against these insects, showing that each solvent used completely removes the insecticidal principle.

Table 48 shows the solvents used, the total amount of extracted material removed by each solvent, and the amount of insoluble resin separating out in each case.

TABLE 48.—*Amount of material extracted by various solvents from the flowers of C. cinerariæfolium (sample No. 26962)*

Solvent	Total extract	Insoluble resin
	Per cent	*Per cent*
Chloroform	8.93	0.04
Carbon tetrachlorid	6.51	.80
U. S. P. ether	7.85	.77
Petroleum ether	3.77	.37
Carbon disulphid	4.57	.62
Methyl alcohol	28.78	11.38
Ethyl alcohol (absolute)	19.18	1.12
Ethyl alcohol (95 per cent)	30.91	4.18
Acetone	16.66	2.20
Benzol	5.74	.00
Ethyl acetate	15.48	1.30

No insoluble resin is formed in extracting with benzol, and only a trace with chloroform. The large amount of insoluble extract in the case of methyl alcohol is explained by the fact that the solution went nearly to dryness through loss of the solvent by evaporation.

In view of the fact that all the solvents completely removed the insecticidal principle, as shown by practical tests, and petroleum ether extracted the smallest amount of material, it was decided to use petroleum ether as the solvent in further work and to use a percolator in making the extractions, to avoid heating the material in solution.

For the percolation a quantity of the highest grade closed Japanese insect flowers procurable was coarsely powdered, packed in a glass percolator and macerated with the solvent for 48 hours. The percolation was then allowed to proceed slowly, the solution being drawn off at the rate of about 1 drop a second. The beautiful slightly greenish-yellow solution obtained was placed in large glass crystallizing dishes, and the solvent allowed to evaporate at room temperature in a current of air. The residue was partly solid and partly an oily liquid, at ordinary temperature, of a reddish-yellow color, and had a strong characteristic odor. Nothing crystalline was observed in this residue. Careful tests showed that it did not contain any nitrogen.

Procedure 1.—For further separation of the materials present in this residue, the entire material extracted by the petroleum ether was saponified by boiling with alcoholic potash (22 grams of potassium hydroxid and 600 cc. 95 per cent alcohol to 32 grams of extract). The boiling was continued for 75 minutes, the alcohol removed by evaporation on the steam bath, the residue taken up in water, and the alkaline solution extracted with ether. The ethereal extracts, which were of a beautiful reddish-yellow color, were combined, washed with a little water, dried over calcium chlorid, and the ether removed by evaporation at room temperature in a current of air. A mass of reddish-yellow powder was left. On recrystallizing this powder from alcohol, the first recrystallization removed all of the red color, and the second all but a trace of the yellow color, leaving crystals of a

very pale yellow. The melting point of these crystals (167° to 168°) and tests for a phytosterol showed it to be a phytosterol-like substance. Tested upon insects (roaches, ants, and aphides), it proved to be inert.

The mother liquors from the recrystallizations were evaporated to dryness in a vacuum desiccator over sulphuric acid. More of the phytosterol-like substance was obtained, but nothing else. This was also found to be inert against the insects mentioned. The insecticidal principle of Pyrethrum flowers is not, therefore, in the nonsaponifiable portion of the petroleum-ether extract.

The solution from the saponification after extraction with ether was made slightly acid with sulphuric acid, and again extracted with ether. These extracts were very dark red, almost black. After washing with a little water, drying over calcium chlorid, and evaporating the ether at room temperature in a current of air a sticky resin was left. Special tests were made for phenols in this material, but the results were negative. Tests on small portions with various solvents failed to yield any crystalline product.

The whole of the saponifiable portion was then dissolved in U. S. P. ether, and about twice its volume of petroleum ether added. This precipitated a very dark, sticky resin, which was filtered off. Tested upon aphides, this resin proved to be inert.

The filtrate from this resin, which contained the fatty acids, was evaporated to dryness, leaving a soft, yellowish, oily, sour-smelling residue. This material was shown to contain acetic and other fatty acids, together with a pungent-tasting oily substance, which was present in too small a quantity for further investigation. When sprayed upon aphides feeding on nasturtium plants, the fatty acids injured the leaves of the plants, but the aphides were unaffected.

These tests show that saponification with alcoholic potash produces a chemical change which destroys entirely the insecticidal action of the material. That saponification with alcoholic potash effects chemical decomposition in some of the compounds present is also shown by the fact that petroleum ether added to the U. S. P. ether solution of the saponifiable portion precipitates a resin, whereas all the material before saponification was soluble in petroleum ether.

Procedure 2.—Since saponification with alcoholic potash destroys the insecticidal action of the material extracted by petroleum ether, another procedure was adopted. A petroleum-ether extract was obtained as before, evaporated to dryness, and taken up in U. S. P. ether. This ethereal solution was successively extracted with aqueous solutions of ammonium carbonate, sodium carbonate, and sodium hydroxid made up on the basis of 10 grams of salt to 100 cc. of solution. The ammonium carbonate solution assumed a light yellow color when shaken with the ethereal solution of the extract. Emulsions which were difficult to separate formed. After repeatedly extracting with ammonium carbonate and washing with water, the ethereal solution was separated and reserved for the sodium carbonate extraction.

The ammonium carbonate solution was made acid with sulphuric acid and shaken with ether, which slowly and apparently incompletely took out the yellow color. On evaporating this ether extract to dryness, after washing and drying over calcium chlorid, a few greenish-yellow, oily, sticky drops were left. From 1,920 grams of

powdered flowers only 0.345 gram of material was thus obtained. This quantity was too small for purification and chemical testing, and was, therefore, used for testing on insects. It was dissolved in about 50 cc. of 95 per cent alcohol, diluted with an equal quantity of water, and sprayed on nasturtium plants which were infested with aphides. From 75 to 80 per cent of the aphides were killed. In check tests with 50 per cent alcohol no aphides were killed.

The aqueous solution of sodium carbonate removed a noticeable quantity of a brownish-yellow material, which was obtained as a sticky mass after acidifying and extracting with ether. Tested against aphides in the same way, the material proved to be very active, killing 100 per cent. Extraction with sodium hydroxid solution removed a quantity of chlorophyll and also some of the insecticidal constituents. About 80 per cent of the aphides sprayed with an aqueous alcoholic emulsion of the extracted material were killed. On evaporating the ethereal solution of the original petroleum-ether extract after the successive extractions with aqueous ammonium carbonate, sodium carbonate, and sodium hydroxid, a strong reddish-orange pasty material was left. Tested on aphides, this material likewise exhibited marked insecticidal power, killing about 90 per cent of the insects.

These tests show that the active principle can not be wholly acidic or phenolic; otherwise all would have been extracted by the aqueous sodium hydroxid solution.

Procedure 3.—The petroleum-ether percolate was not evaporated to dryness, but was first extracted with a saturated aqueous solution of sodium bisulphite to remove aldehydes, if present, and then with 1 per cent sodium hydroxid.

Acidifying the sodium bisulphite extract with sulphuric acid, extracting with ether, and evaporating to dryness, gave a trace of a sticky varnish. This had a pleasant odor, gave a somewhat indefinite test for aldehydes, and appeared to be a polymerization product. Too little was obtained for satisfactory testing. Apparently, however, a very small quantity of aldehyde is present in the petroleum-ether extract of insect flowers. The sodium hydroxid extract contained some chlorophyll, and in other respects closely resembled the sodium carbonate and sodium hydroxid extracts obtained in procedure 2.

The residual petroleum-ether solution, evaporated to dryness, after successive treatments with the bisulphite and hydroxid of sodium, left a sirupy, reddish material similar to the residue obtained in procedure 2.

Identification tests on the materials isolated in procedures 1, 2, and 3.—The extracts were subjected to several special tests as outlined in Mulliken's "Identification of Pure Organic Compounds" and other standard works. The results showed that the petroleum-ether extract of the flowers of *C. cinerariæfolium*, which completely removes all substances of insecticidal action, contains no phenols and no nitrogen-bearing compound, but that it consists largely of an ester or esters, together with a trace of aldehyde and a quantity of free acids.

These results harmonize some of the discrepant conclusions reached by the previous investigators, especially Schlagdenhauffen and Reeb, who claimed that the active principle is an acid, to which they gave

the name of pyrethrotoxic acid, and Fujitani, who claimed that it is a neutral ester, which he named pyrethron.

Work Done from 1923 to 1924[8]

In 1923 work on Pyrethrum was resumed with the object of isolating the active principle in pure condition. About 2 kilos of a coarse powder ground from whole Dalmatian flowers was extracted in a glass percolator, using petroleum ether boiling between 20° and 45° C. as the solvent. Decidedly less extracted material was obtained when light petroleum-ether was used (about 1.8 to 2.5 per cent). The oily product obtained by evaporating off the solvent was poured into a large volume of 95 per cent alcohol, which caused the precipitation of a waxy material. The alcoholic solution was allowed to stand in the cold room at a temperature of about −5° C. for 24 hours, which caused the separation of more fats and waxes. The solution was filtered in the cold and the alcohol evaporated in vacuum. The resulting thick sirup was subjected to distillation in a vacuum of 1 to 2 millimeters, using small quantities of material for each distillation. Foaming caused some trouble, but careful operation made it possible to distil over about 20 per cent of the material. The distillate was collected between 100° and 190° C. The temperature of the oil bath ranged from 140° to 230° C. The distillate was a light yellow oil, which partly solidified on cooling to a mass of about the consistency of butter.

By direct saponification of this crude distillate with alcholic potash, the pyrethronic acid of Yamamoto (*S32*) was obtained and converted into the dihydroxy acid, which was recrystallized from a mixture of acetic ether and petroleum ether. It melted at 146°–147° C., and the analytical results agreed with those reported by Yamamoto.

In order to remove the free acids, the crude distillate was dissolved in alcohol and treated with a solution of lead acetate. This gave the lead salt of an acid, from which the free acid was isolated and purified by recrystallization from alcohol. It was identified as palmitic acid.

The alcoholic solution was evaporated, the residue taken up in ether and extracted with water, and the solvent evaporated. In later experiments the first distillates were dissolved in ether and extracted with dilute alkali and the ethereal solutions were dried with sodium sulphate. The residue from the ethereal solution was subjected to repeated fractionation in a vaccum of about 2 millimeters. The material distilling up to 150° C. was discarded and the main portion of the distillate boiling between 150° and 166° was collected separately and again distilled. The product boiling between 153° and 166° was analyzed, with the following results: Carbon, 74.20 per cent; hydrogen, 9.26 per cent; molecular weight, 293 by the freezing method; density at 20° C., −12.84°. Several preparations obtained by this method and analyzed showed only slight deviations from the values given. From the analytical figures the formula $C_{18}H_{26}O_3$ was calculated. Determinations of the molecular weight by saponification, however, showed a slightly higher value than that found by the freezing point method, indicating the presence of 5 to 10 per cent of unsaponifiable material. Different preparations and different fractions of the same preparation taken at only slightly different temperatures also showed varying refractive index values.

[8] Conducted by F. B. LaForge.

These results indicated that the material, although very nearly pure, was contaminated with other compounds of about the same boiling point as the toxic substance. The yields of redistilled material generally represented 0.1 to 0.15 per cent of the powder.

The preparations were highly toxic to *Aphis rumicis*, killing the insects in dilution up to 1 to 10,000.[9]

Great difficulties were encountered in attempting to isolate the products of saponification of the material in the case of the alcoholic component, which seemed to decompose under all conditions.

While this phase of the work was in progress, a series of 10 articles on the toxic principles of insect powder was published by Staudinger and Ruzicka (*S28*). Had these articles, describing results obtained from 1910 to 1916, appeared before the bureau's investigations were begun, all this work on the constitution of the active principles of Pyrethrum would have been unnecessary. These investigators had isolated the toxic principles and had practically solved the problem of their chemical nature.

Staudinger and Ruzicka used the following method: The powdered flowers were extracted with light petroleum ether. The extract was evaporated and the residue was extracted repeatedly with methyl alcohol, which removed the toxic substances, together with fatty acids and other impurities. By strongly chilling the alcoholic solution, a large part of the impurities was removed. The methyl alcohol was expelled and the residue taken up with petroleum ether. Fatty acids were extracted from the solution by shaking with potassium carbonate solution and transforming the potassium soaps into calcium soaps by addition of calcium chlorid. This treatment was necessary in order to avoid the formation of emulsions. The petroleum ether solution was evaporated and the residue extracted with methyl alcohol, which solvent was in turn removed and the resulting product again dissolved in petroleum ether and the solution once more treated with potassium carbonate solution. Upon evaporation of the solvent, a crude product was obtained in quantity corresponding to 0.47 per cent of the powder. This consisted of about 50 per cent of the active substances. By distillation of this crude product in a high vacuum, a highly active oil was obtained. It was not possible, even by repeated distillation, to completely purify the material, however. Treating the distilled material with semicarbazid in methyl alcohol gave a semicarbazone, which could be recrystallized and thus prepared in nearly pure condition. By hydrolysis with oxalic acid, a practically pure compound was prepared. This compound, represented by the formula $C_{21}H_{30}O_3$, was named pyrethrin I. It is an ester of a ketone-alcohol and is highly toxic to insects. The semicarbazone can be hydrolyzed with methyl alcoholic-sodium hydroxid into the semicarbazone of the ketone-alcohol and an acid.

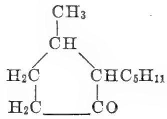
The acid which Staudinger and Ruzicka named chrysanthemum acid is the same as the pyrethronic acid of Yamamoto, and its constitution was determined by oxidation to transcaronic acid and acetone by means of ozone. It is represented by the structural formula

$$
\begin{array}{l}
(CH_3)_2\,C{-}CHCH{=}C(CH_3)_2 \\
\quad\;\; \diagdown\;\diagup \\
\qquad HC \\
\qquad\;\; | \\
\qquad COOH
\end{array}
$$

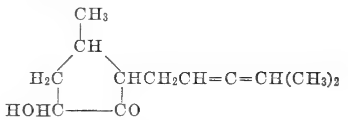
[9] Toxicity tests were conducted by C. H. Richardson, Bureau of Entomology.

This formula was confirmed by synthesis of the racemic form. The semicarbazone of the ketone-alcohol is best hydrolyzed by long shaking in benzol solution with an aqueous solution of potassium bisulphate. The free alcohol can then be purified by distillation in a high vacuum. It has been named pyrethrolon.

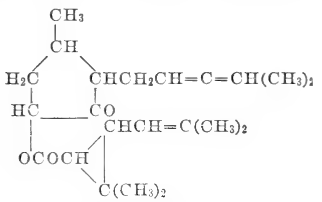
The constitution of this ketone-alcohol was worked out from its products of oxidation and reduction and by synthesis of the fully reduced tetrahydropyrethron obtained by reduction with hydrogen in the presence of platinum black. Tetrahydropyrethron was shown to have the formula



and pyrethrolon the formula



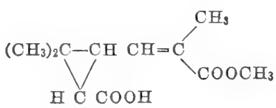
The acid chlorid was prepared from chrysanthemum acid and combined with pyrethrolon to form pyrethrin I. Although neither the acid nor the alcohol showed any toxic properties, the resynthetized ester showed the same toxicity as the original pyrethrin. Thus pyrethrin I may be represented by the formula



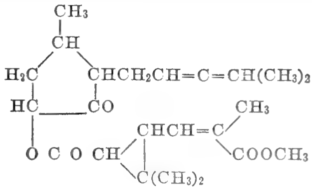
Treating the crude undistilled oil with semicarbazid gave an impure mixture of two semicarbazones, which it was not practical to separate. The mixture was therefore saponified with methyl alcoholic-sodium hydroxid solution, by which treatment pyrethrolon semicarbazid, together with a mixture of acids, was obtained. More of these products was obtained by saponification of mother liquor from the first crystallization. It was not possible to prepare the second semicarbazone directly.

From the mixture of acids, chrysanthemum monocarboxylic acid and two other acids (chrysanthemum dicarboxylic and chrysanthemum dicarboxylic-acid-mono-methyl-ester) were obtained. When treated with ozone chrysanthemum dicarboxylic acid yielded transcaronic acid and levulinic acid. Its methyl ester yielded transcaronic acid and levulinic acid methyl ester.

From these results it follows that chrysanthemum dicarboxylic acid methyl ester must have the structural formula



This acid was converted into the chlorid and combined with pyrethrolon to form pyrethrin II, which is represented by the formula



Thus the pyrethrins are esters of pyrethrolon with chrysanthemum monocarboxylic acid and chrysanthemum dicarboxylic acid methyl ester. The toxic principles of pyrethrum consist of about 40 per cent of pyrethrin I and 60 per cent of pyrethrin II.

The foregoing formulas contain five asymmetric carbon atoms and are highly unsaturated. Owing to their extremely complicated nature, there is little or no hope for the technical synthesis of pyrethrin I and II.

Because of the possibility that less complicated compounds containing some essential grouping might have properties similar to those of the natural products, a large number of esters in which pyrethrolon was combined with various acids and in which various alcohols were combined with the chrysanthemum acids were prepared. In a very few cases the resulting products were toxic, but in no case did the toxicity approach that of the natural pyrethrins. Even when an acid differing from chrysanthemum acid by having one methyl group replaced by hydrogen was combined with pyrethrolon, the product was only about one-eighth as toxic as the natural product and synthetic pyrethrin containing inactive chrysanthemum acid was very much less active. The reduction of one or more double bonds in either the acid or the alcoholic component completely destroyed the toxicity. Compounds of the chrysanthemum acids with bases were completely inactive. There seems therefore to be little prospect of obtaining commercial synthetic insecticides related to the pyrethrins.

DISTRIBUTION OF THE ACTIVE PRINCIPLE IN CHRYSANTHEMUM CINERARIÆFOLIUM

It has long been known that the flowers of the *Chrysanthemum cinerariæfolium* possess insecticidal properties. The action of the stems is so slight that they are practically worthless as an insecticide. It has been generally supposed that the buds, or "closed" flowers, are more active than the mature or "open" flowers. The trade formerly divided insect flowers into three grades: "Closed," "half-closed," (or "half-open"), and "open." The "closed" flowers usually sold for about twice as much as the "open" flowers, which would imply that their insecticidal power is correspondingly greater.

Gerard (*93*) is the only investigator who states in what part of the flower the active principle is chiefly found. According to him there are two active substances, an oleoresin and an essential oil, which are found principally around the ovaries of the flower, and to a small extent in the corollas.

The work on insect flowers and stems here reported, in which it is shown that the nitrogen content increases with the grade of the product, led to a determination of this constituent in the different parts of the flower. At the same time a test on insects was made to see if the insecticidal effect of these parts was related to the quantity of nitrogen present. Typical commercial "open" flowers (*C. cinerariæfolium*) were dissected into their principal parts. In most commercial samples, the greater number of the flowers have lost their disk and ray flowers, and consist only of fruit, receptacles, and involucral scales. The fruit amounted, on the average, to 80.5 per cent, the receptacles to 10.8 per cent, and the involucral scales to 8.7 per cent of the whole flowers. Enough disk flowers were collected to determine their nitrogen content, but these were lacking in so many of the "open" flowers that their natural relative proportion could not be determined. Their normal actual percentage by weight, however, is very small. The results of the analyses are given in Table 49.

TABLE 49.—*Determination of nitrogen content of insect flowers*

Sample	Nitrogen	Percentage of total nitrogen
	Per cent	
Entire "open" flower	1.26	
Fruit	1.40	89.4
Receptacles	.67	5.7
Involucral scales	.51	3.5
Disk flowers	1.68	

The nitrogen in the fruit of five other samples of "open" flowers was also determined, with the results shown in Table 50.

TABLE 50.—*Nitrogen in fruit and flowers of insect flower plants*

Fruit	Entire "open" flower
Per cent	*Per cent*
1.34	1.31
1.32	1.22
1.34	1.28
1.24	1.25
1.26	1.25

These results show that the fruit from commercial samples of "open" flowers contains about 90 per cent of the total nitrogen.

E. W. Scott and W. S. Abbott, of the Bureau of Entomology, conducted practical tests on roaches to determine the relative insecticidal strength of different parts of the finely powdered flowers. The roaches were dipped in the powder until all parts of the body were covered with it, and then placed in 8-ounce bottles, 1 insect to a bottle, 10 insects being used in each test. All of the insects were allowed ventilation, but no food or water. The average of the 10 tests for each powder is given in Table 51.

TABLE 51.—*Insecticidal strength of finely-powdered parts of insect flowers*

Sample No.	Source	Time required to paralyze	Time required to kill
		Minutes	*Hours*
1	Entire "open" flowers of *C. cinerariæfolium*	3.7	21
2	Fruit from sample 1	2.4	19½
3	Ray flowers from sample 1	1,800	63
4	Receptacle from sample 1	90	50½
5	Involucre from sample 1	240	80
6	Disk flowers from "closed" flowers of *C. cinerariæfolium*	8	31½
7	Stems		131
8	Flowers of *C. roseum*	7	23
9	Check		236½

Apparently the fruit and disk flowers of *Chrysanthemum cinerariæfolium* are the parts most active in paralyzing and killing roaches. These parts show the highest content of nitrogen, so that this element, although not actually a constituent of the active principle, appears to be present with it in a constant ratio, and hence can be used as a measure of the insecticidal activity.

Summary of work on the active principle.—All common organic solvents completely remove the insecticidal principle from the flowers of *C. cinerariæfolium.* The insecticidal principle is not removed by a steam distillation. Water and dilute acids do not dissolve it, but dilute potassium hydroxid solution removes all of the activity from the powdered flowers. The active principle consists of two closely related complicated esters (pyrethrin I and pyrethrin II), present in the flowers to the extent of 0.2 to 0.3 per cent. The chemical nature of these two compounds is now well understood. Slight changes in their chemical structure greatly decrease their toxicity and in most cases destroy it completely. There seems to be little prospect of their commercial synthesis or of obtaining toxic compounds related to them but of less complicated structure.

SUMMARY

The fact that flowers of certain species of Pyrethrum have the property of killing various insects was known to the eastern Europeans more than a century ago. Since then this knowledge has gradually spread, until insect powder is now a common household convenience. This powder owes its insecticidal activity to a mixture of esters, the chemical nature of which is now well understood. These esters first benumb and then kill the insects brought into contact with the powder. Although generally considered to be harmless to the higher animals, it has produced symptoms of a more or less serious nature in a number of cases, according to the records in the literature.

Insect flowers are now cultivated commercially in Dalmatia, Japan, Australia, France, Algeria, and California in the United States, the first two countries producing nearly all of the flowers that enter into international trade. The powder is made in each of these countries.

In the enforcement of the insecticide act, insect powder has been found adulterated in a variety of ways. In some instances such substances as lead chromate, curcuma, and yellow ocher are added to give color. Other species of flowers, like the Hungarian or ox-eye daisy, are substituted in whole or in part for the true insect flower. Almond

shells, brick dust, hellebore, pepper, sawdust, starch, sumac, and the like have been found less frequently in samples examined. The ground stems of the Pyrethrum plant and powdered ox-eye daisy flowers constitute probably more than 90 per cent of the adulterants used in insect powder at this time.

Physiological, chemical, and microscopical methods which can be used satisfactorily in detecting adulteration with powdered stems and ox-eye daisy flowers have not yet been perfected to such a degree as to make an accurate quantitative determination possible. However, from the data obtained in the examination of hundreds of samples of genuine insect powder, of the materials used for its sophistication, and of commercial samples, the results of which are reported in this bulletin, it is possible to determine in an insect powder the approximate quantities of these adulterants if present.

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